

METALLURGIA

The British Journal of Metals

(INCORPORATING THE METALLURGICAL ENGINEER)

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PRINCIPAL CONTENTS IN THIS ISSUE:

	Page
Industrial Research	253
The War and Shipping	254
The London Metal Exchange	254
Copper-Lead Bearings by Powder Metallurgy. By W. D. Jones, M.Eng., Ph.D.	255-260
<i>The results of an investigation, which is described in this article, indicate the simplicity and advantages of manufacturing copper-lead bearings, in the form of bushings or bonded to a steel backing, by the technique of powder metallurgy.</i>	
The Analysis of English Fluorspars and their Associated Minerals. By G. H. Osborn	261-264
<i>The increasing use of home-mined fluorspars for metallurgical purposes has imposed a strain on English mines, few of which have flotation and washing plants to obtain the necessary grades.</i>	
Cerium and Some of Its Applications. By L. David	264-265
<i>In the form of cerium standard alloy, the best known use of cerium is in the production of flints for spark-emitting instruments.</i>	
Gas and Air Flow in Gas-fired Furnaces	266
Metallurgical Coke. By R. J. Barritt, M.A.	267-272
<i>The use of coke as fuel led to greatly increased sizes of blast furnaces, which in turn involved development of coke possessing suitable properties.</i>	
Progress in Micro-Testing. By D. W. Rudorff, M.Inst.F.	273-277
<i>Present-day testing methods are criticised by Chevenard, and this review of his recent work on the subject will be of interest.</i>	
A Thermodynamical Theory of Restoration Phenomena in the Ageing of Copper-Aluminium Alloys	278

	Page
Research in the Ferrous and Non-Ferrous Industries	279-280
<i>Research is a vital necessity for the maintenance of British industry at a high level, and attention is directed to the organisation of co-operative research associations.</i>	
The British Non-Ferrous Metals Research Association. By G. L. Bailey, M.Sc.	280-283
The Iron and Steel Industrial Research Council	284-286
The Iron and Steel Institute	287-288
<i>Annual Autumn Meeting in London.</i>	
A Study of Austenitic Grain Growth in Medium-Carbon Steels. By J. H. Whiteley	288-290
New Phosphate Coatings with Unusual Corrosion Resistance	290-291
<i>An investigation of a pre-dip solution is outlined, and a pre-dip containing 1-2% disodium phosphate, activated with a titanium compound, is described.</i>	
An Investigation of the Effects of Precipitation Treatment on Binary Magnesium-Aluminium Alloys. By F. A. Fox, M.Sc., and E. Lardner, B.Sc.	293-298
<i>Close metallographic examinations have been made of binary magnesium-aluminium alloys containing 4, 6, 8, 10 and 12% aluminium in the solution-treated and precipitated state. The effects of time and temperature have been studied and four general forms of precipitate are discussed.</i>	
Improved Temperature Regulators	298
Radiography of Metals and Alloys	299-302
<i>Review of an American symposium.</i>	
Microchemical Analysis	303-304
Converting Furnaces from Oil to Coal Firing	305-307

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METALLURGIA

THE BRITISH JOURNAL OF METALS.
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OCTOBER, 1943

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Industrial Research

THE labour of a host of men, of great laboratories and long patient research, build up the sound structure of knowledge and contribute to progress. Sometimes the building proceeds brick by brick, but often progress appears slow, until the addition of fact to fact culminates in a revolutionary discovery. Systematic exploration avoids confusion, reduces the possibility of premature conclusions, indicates the often unexpected influence of one factor upon another and results in the final application of a discovery or development for the greatest benefit of mankind. It is this research which is the essential basis of industrial progress and which is going to be the vital factor in determining the future prosperity of Great Britain.

More than ever before, as Sir Harold Hartley, F.R.S., points out in a recent pamphlet, the prosperity of Britain will depend upon the efficiency and progressiveness of our industries. The loss of our foreign investment and the possible diminution of the payments to us from abroad for services rendered will necessitate a considerable expansion in the value of our exports if we are to increase or even maintain our standard of living. Furthermore, this increase in exports will have to be brought about despite the industrialisation of other countries which, before the war, were mainly producers of raw materials. Success can only be won if our products are better, more attractive or cheaper than those made by our competitors or in our customers' own country. In the home market also our own products must hold their own, and we must make the most of the limited range of our native raw materials. Every business man should realise why the need for industrial research is so urgent for Great Britain.

This formidable task can only be achieved by using to the full our inventiveness and technical skill both to increase the efficiency of our older industries and to develop new commodities which will hold their own in the markets of the world. Our natural genius and craftsmanship gave us industrial supremacy in the nineteenth century, at the close of which we had few competitors. Then, of course, we were cultivating a virgin field, to-day, however, the position is very different since what might be regarded as the obvious developments have already been made and further secrets are now only revealed as a result of much more prolonged and careful searching. Haphazard inquiry must be replaced by organised and systematic study. Only research can refashion existing industries effectively and create new ones. Certainly, in the future, the advantages Britain possesses in the skill and

traditions of her craftsmen will depend more and more on the science that directs their efforts. Labour and research must work hand in hand.

Much criticism has been directed against the industrial research activities of this country. It is said that in comparison with a number of competitor countries the time and money spent on research is relatively small. Direct comparisons are difficult and misleading in that much valuable work has been done in the industries of this country without it being specially termed industrial research. Apart from outstanding exceptions, however, much of the criticism is justifiable, since, in general, there appears to be a lack of appreciation of the value of science in industry and the amounts spent on research are correspondingly less. But it should be emphasised that this position is not due to any lack of ability; in both the world wars British scientists have proved that they can more than hold their own if they are given equal facilities and support. All they need is the same opportunity in peace as in war, and removal of the apathy which seems to exist amongst a great number of men in particular industries. If the men and firms concerned could be persuaded to become research-minded, ways and means would quickly become available to meet requirements. It is true that the Government can do much, but industry must awaken to the need for concentrated effort. There is danger in delay, since markets once lost are not easily regained, active steps should therefore be taken to organise now.

At present, research in Great Britain is carried on in five main groups of establishments: laboratories of the Universities and Technical Colleges; research stations of the Department of Scientific and Industrial Research, and the Agricultural and Medical Councils; research associations supported partly by industry, but aided by grants from the Department of Scientific and Industrial Research; research laboratories maintained by private firms; and research establishments of the Fighting Services. Elsewhere in this issue reference is made to research associations within our field, that are supported partly by industry.

In pure science, and in its application to the arts, research is one of the most potent influences to progress, because it is the application of the results of organised research that gives improvement in materials, machines, processes, and in understanding. To maintain this country's position in the world markets, our industries must greatly increase their attention to research as soon as the progress of the war makes it possible. Whatever else we do, whatever advantages we possess, research is the key to the future and there should be no delay in organising it fully.

The fact that goods made of raw materials in short supply owing to war conditions are advertised in "Metallurgia" should not be taken as an indication that they are necessarily available for export.

The War and Shipping

SHIPBUILDERS, ship-repairers and marine-engine builders are directly concerned with the most vital and urgent task for Great Britain in the present struggle—the maintenance and development of sea power—and, because of the urgency of this task, they have been the target for almost as many criticisms as have those in more exalted positions and often from equally informed quarters. We had occasion to refer to this fact some months ago when we discussed the question of faster cargo ships. It was emphasised at that time that in normal times the economical speed of a cargo ship depends upon a rather sensitive balance between several factors, such as earnings from the cargo carried, loading and discharging times, and cost, running and capital charges, insurance, etc. Under war conditions, other considerations are obviously involved, but the adoption of revolutionary changes in design in such an emergency absorbs a tremendous amount of time before production can proceed at the desired speed. At the outbreak of the war the average speed of cargo vessels would not exceed 11 knots, obviously, therefore, the convoy system had to be built up with vessels that were available, and these had to be supplemented with new ships as fast as shipyards could construct them.

The most dangerous chink in the armour of this country has been the vulnerability of its vital sea-lines of communication and supply. It is noteworthy, therefore, that Mr. J. Ramsey Gebbie, O.B.E., in his presidential address to the North-East Coast Institution of Engineers and Shipbuilders, should refer to the controversy. Criticism of the policy of producing ships of moderate speed in the largest numbers succeeded the most fulsome praise for the vigorous policy which had been initiated by Britain, by which such ships had been turned out in American yards in quantities sufficient to neutralise the depredations of the enemy U-boats. As Mr. Gebbie states, no construction proposals for the production of adequate numbers of sufficiently faster ships by a soon-enough date were offered, but fantastic proposals received much publicity. In this there was much of what the Americans term "hind-sense," or wisdom after the event, but all the while the Admiralty was more realistically conscious of the dangers of the situation than any of the critics, and measures have been taken which have greatly mitigated the danger, while opportunity has also been found to increase the number of faster ships for services in which such vessels can be advantageously employed. Mr. Gebbie is convinced that if we had concentrated on building faster ships during this war, with the inevitable result that a much fewer number would have been available, our recent enormous amphibious operations might have been impossible; even a 15-knot ship can only be in one place at one time, and two 11-knot ships are obviously of much greater value under some conditions than one 15-knot ship. Mr. Gebbie had no intention of suggesting that criticism should not be made, but considered it could only be of value when based on that attention to detail and calculation which shipbuilders and engineers are accustomed to apply when designing ships and engines.

It is generally accepted by experts that to have embarked on the construction of faster ships to replace losses, would have involved considerable reorganisation of the shipyards and engineering works at a time when delay would have been suicidal.

The London Metal Exchange

What is to become of it?

SINCE early in the war the commodity markets have ceased to function as they did in peace time, and the question is frequently asked: What is to become of them after the war? This question was raised by Mr. J. D. Wolff, of the London Metal Exchange, recently, at a luncheon given to mark the close of his 15 years' chairmanship of the Committee of that market at which he made a convincing case for the return of a free and open market in non-ferrous metals.

Viewing the future, Mr. Wolff expressed his conviction that the London Metal Exchange, in common with other commodity markets, should be re-established as soon as possible after the close of hostilities. One of the alternatives to a free market would be a system in which prices continued to be fixed, and supplies guaranteed by the Government. This he condemned as impracticable short of complete international agreements which would not merely guarantee supplies at fixed prices, but offer guaranteed markets to the outside world and fixed selling, as well as buying, prices. Moreover, Government trading would inevitably generate international political friction, since each trading operation would, under such a system, acquire a political significance, which is wholly absent when business is transacted in a free and open market.

The other alternative to the restoration of an open market would be to have prices fixed by agreement between producing interests, strengthened, in all probability, by a further agreement for the control of output. Apart from the more general objections to such restrictive devices, Mr. Wolff considers that the monopoly prices determined by this system would hardly inspire widespread confidence. Moreover, under the Cartel system, producers might be compelled to become bull speculators in their own produce.

It is claimed that the London Metal Exchange avoided these defects. It provided a market where large tonnages could be bought and sold at any time, whether for immediate or forward delivery. It was a market where the copper and tin producer could sell his metal as soon as he had mined the ore from which it was to be produced; perhaps months before it reached the country to which it was assigned. The point is also emphasised that it provided facilities by which the manufacturer could make sure of the price at which he would buy raw material needed months ahead and by which he could hedge the speculative risk he ran by holding stocks of raw materials.

There can be no doubt that the London Metal Exchange has done useful work, but the maintenance of stable prices for the basic materials consumed is a potent in the success of the engineering, shipbuilding and manufacturing industries. It is true that many factors influence metal price movements in normal times and stabilisation is difficult, but considerable fluctuations greatly increase the manufacturers' problems. In 1936, for instance, the difference between the highest and lowest prices for copper was £14 18s. per ton, and the price of lead showed a difference of £9 13s. 9d. Thus, we believe that consumers would welcome some scheme which would bring about the stabilisation of basic raw materials at an economic figure.

Copper-Lead Bearings by Powder Metallurgy

W. D. Jones, M.Eng., Ph.D.

The copper-lead bearing alloys present peculiar casting difficulties, and attention is directed to the manufacture of these alloys, in the form of bushings or bonded on to a steel backing, by the technique of powder metallurgy. The results of an investigation, which is described, indicate the simplicity and advantages of approaching this class of bearing by this method.

Introduction

ADVANCES in the design of internal combustion engines particularly stimulated by progress in aeroplane engines has caused an increasingly insistent need for bearing metals that will withstand higher pressures and temperatures than do the ordinary tin-base alloys, which were formerly entirely satisfactory. The copper-lead bearing materials, either alone or bonded on to a steel backing, are at the present time fulfilling this function, and are in fact considered by many people as being the only bearing materials capable of withstanding the severe loading and peripheral speeds without deterioration of physical properties on temperature rise customary in the present-day ignition engine. In war time these alloys are also of some interest because of the necessity of reducing the consumption of tin.

These alloys present peculiar casting difficulties, and reference to the copper-lead constitutional diagram shown in Fig. 1 provides an obvious explanation, as follows:—All compositions freeze by the separation of copper dendrites, and every alloy will contain a proportion of lead which is molten down to the freezing point of lead at 327° C. This is a very extensive cooling or plastic range, and it is obvious that unless the cooling rate is extraordinarily rapid serious segregation and agglomeration of the lead globules is bound to occur. With compositions containing more than some 80% of copper the primary dendrites of copper are sufficiently extensive in volume to render this casting difficulty less serious. It will be noted, however, that with compositions exceeding 36% of lead the liquid phase separates into two immiscible compositions, and marked undesirable segregation is bound to take place even before any solidification commences. This really complicates matters and affords the principal explanation why the lead content of this type of alloy is restricted to 36% of lead unless very special foundry techniques are employed.

Various manufacturers of these materials employ special foundry techniques which avoid these segregation difficulties with varying degrees of success, but apart from special casting techniques, much can be done by composition adjustments. This is regularly employed for preventing segregation of lead, and alloys of this type frequently contain up to 15% of tin. Relatively small percentages of other additions are very helpful in this way, and small additions of sulphur, and of nickel to to 2.5%, are very well known in this connection.

The idea of approaching the manufacture of these alloys either in the form of bushings or bonded on to a

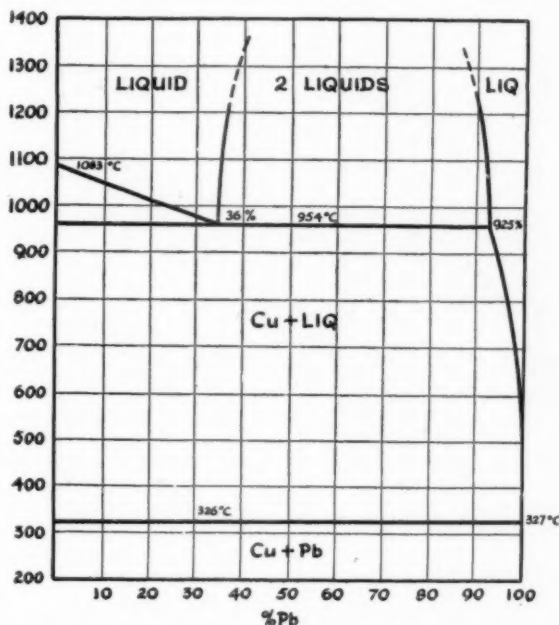


Fig. 1.—The copper-lead constitutional diagram.

steel backing by the technique of powder metallurgy is comparatively recent, although it is a very sound direction in which to progress, because not only is it possible to obviate completely all these segregation difficulties, as will be shown in this paper, but it is a very great advantage to be able to make use of some of the obvious advantages of the powder metallurgy technique—namely, complete avoidance of inhomogeneity, blowholes, casting difficulties, etc., and particularly the extensive waste of metal in the form of risers, headers, gates, etc., associated with all castings.

Preliminary experiments with copper and lead powders have shown, however, that it is not particularly simple to press and sinter such mixtures, because at any reasonable sintering temperature the lead melts and exudes, and at any lower temperature the cohesion of the copper particles is not sufficiently substantial. Various devices have been suggested to overcome this trouble. Patents have been filed, for example, covering the coating of each particle of lead with copper by electro-deposition, and there are a number of other suggestions along these lines.

One method which, however, has already achieved considerable commercial success consists of taking the molten mixture of the two metals above the temperature of immiscibility, whatever that may be in view of the composition, and atomising the melt to form a powder. It is clear that not only is there no limitation of composition to 36% of lead by this technique, but that the rate of cooling of a fine globule or a fine particle of powder is so rapid that no segregation of lead is possible.

Microscopic examination of powders containing less than 50% lead made by this process shows that each particle is constituted by a continuous phase of copper enveloping particles of lead, some of which are very fine and some of which are comparatively coarse. From the point of view of pressing and sintering such powders the advantages of this process are:—

- (1) That the lead is entrapped within the copper and cannot exude to any substantial amount whatever the sintering temperature.
- (2) That the lead cannot interfere with the particle-to-particle contact of the copper, and therefore the strength of the sintered copper matrix is not impaired.
- (3) That the distribution of lead within the copper is very good indeed when compared with normal methods of casting these materials.

Each particle can be considered as a fine sample of the main bulk. Fig. 2 shows a photomicrograph of one particle of this type of copper-lead powder under a magnification of 600 diameters.

In order to afford guidance in the technique of pressing and sintering these copper-lead powders both for bushings and for steel-backed bearings the exploratory research work reported here was undertaken by H. W. Jones in the laboratories of F. W. Berk and Co., Ltd., London, upon samples of powders manufactured by them and marketed by Powder Metallurgy, Ltd. It is hoped and believed that the publication of this brief investigation will indicate to bearing metal manufacturers the simplicity and advantages of approaching this class of bearing material in this way.

Description of Research

The powder compositions employed were made up nominally of 18%, 25%, and 40% lead respectively. These on analysis checked 16.5%, 25.5%, and 36.5% lead. For the purpose of this research these powders were sieved through a 200-mesh I.M.M. sieve. The following table indicates the screen analyses, loading weights and flow factors:—

TABLE I.
SCREEN ANALYSES.

	On 30 Mesh.	On 40 Mesh.	On 60 Mesh.	On 100 Mesh.	On 150 Mesh.	On 200 Mesh.	On 300 Mesh.	Through 300 Mesh.
16.5% alloy	—	16.7	10.20	9.00	14.10	10.20	10.40	29.40
25.5% alloy	0.45	—	1.74	4.67	7.56	10.30	39.20	36.00
36.5% alloy	—	6.00	7.20	7.20	8.30	29.00	3.20	39.10

LOADING WEIGHT.

16.5% alloy 4.48 gms./cc.

25.5% alloy 4.09 gms./cc.

36.5% alloy 4.49 gms./cc.

FLOW FACTOR (ORIFICE 0.177 IN.)

16.5% alloy 8.61 gms./sec.

25.5% alloy 10.20 gms./sec.

36.5% alloy 8.33 gms./sec.

PARTICLE SHAPE: Spherical.

It is the purpose of this research to investigate the pressing and sintering characteristics of this powder and to ascertain the optimum pressures and temperatures in order to obtain maximum solidity without

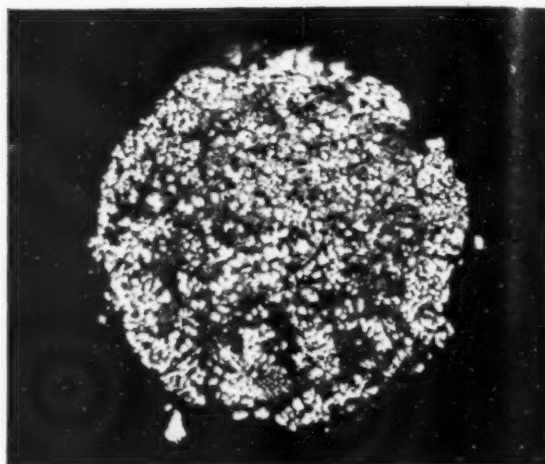


Fig. 2.—One particle of copper-lead powder. $\times 600$.

exudation of the lead and any overdue distortion in shape.

The pressings were made in a pellet die of heat-treated steel $1\frac{1}{8}$ in. I.D. Each pressing was made in duplicate; densities were determined by measurement of the diameter and length of the pressing and its weight. As confusion might arise in an endeavour to interpret the densities because of the varying compositions, the results are reported as percentage solidity calculated on the basis of the analysed compositions.

TABLE II.

No.	% Pb.	Pressure Tons/Sq. In.	Weight.	Dia. in Cm.	Length in Cm.	Volume in Cc.	Density.	% Solidity.
1	16.5	5	4.1328	0.9315	0.870	0.62	6.66	72.0
2	"	5	4.0662	0.936	0.871	0.625	6.5	70.8
3	"	10	4.1444	0.9525	0.7465	0.5315	7.78	81.0
4	"	10	4.1316	0.957	0.739	0.531	7.78	84.1
5	"	20	4.1312	0.958	0.673	0.485	8.525	92.1
6	"	20	4.1428	0.953	0.681	0.485	8.54	92.3
7	25.5	5	4.2044	0.957	0.844	0.607	6.92	73.5
8	"	5	4.179	0.956	0.826	0.595	7.04	74.5
9	"	10	4.2832	0.9515	0.852	0.605	7.06	74.65
10	"	10	4.1956	0.957	0.721	0.5185	8.1	85.7
11	"	10	4.1938	0.957	0.723	0.519	8.06	85.3
12	"	20	4.1884	0.958	0.656	0.572	8.85	93.6
13	"	20	4.1844	0.953	0.653	0.465	8.99	95.2
14	36.5	5	4.3286	0.952	0.854	0.607	7.12	73.6
15	"	5	4.3614	0.9535	0.86	0.613	7.11	73.5
16	"	10	4.2964	0.953	0.752	0.536	8.01	82.8
17	"	10	4.4264	0.953	0.770	0.549	8.06	83.4
18	"	20	4.305	0.959	0.6735	0.485	8.87	91.7
19	"	20	4.2994	0.959	0.6735	0.486	8.84	91.4

TABLE III.

No.	% Pb.	Pressure Tons/Sq. In.	Weight.	Dia. in Cm.	Length in Cm.	Volume in Cc.	Density.	% Solidity.
1	16.5	5	4.098	0.903	0.832	0.532	7.70	83.1
2	"	5	4.019	0.901	0.827	0.5265	7.63	82.5
3	"	10	4.113	0.976	0.749	0.56	7.34	79.4
4	"	10	4.100	0.978	0.740	0.555	7.40	80.0
5	"	20	4.104	0.972	0.731	0.542	7.57	81.9
6	"	20	4.172	1.011	0.719	0.576	7.25	78.4
7	25.5	5	4.178	0.957	0.818	0.588	7.1	75.1
8	"	5	4.135	0.964	0.802	0.585	7.07	74.8
9	"	5	4.167	0.952	0.821	0.585	7.12	75.4
10	"	10	4.1175	0.966	0.742	0.544	7.55	80.0
11	"	10						
12	"	20						
13	"	20						
14	36.5	5	4.3055					
15	"	5						
16	"	10						
17	"	10						
18	"	20						
19	"	20						

Distorted in Sintering.

Distorted in Sintering.

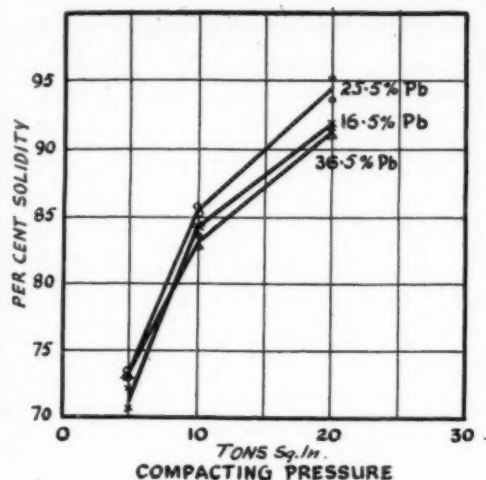


Fig. 3.—Densities of various compositions under different pressures.

The work was commenced by making a number of pressings in the three compositions under 5, 10 and 20 tons per sq. in. pressure. These results are reported in Table II, each specimen having its specific serial number. These results are also shown in Fig. 3. It will be seen that the powder possesses good cold-pressing properties, and that high percentage solidities of 91% to 93% are

TABLE IV.

No.	% Pb.	Pressure, Tons/Sq. In.	Weight.	Dia. in Cm.	Length in Cm.	Volume in Cc.	Density.	% Solidity.
22	16.5	5	4.119	0.9515	0.847	0.602	6.84	74.0 C.P.
22	"	5	4.081	0.917	0.822	0.543	7.51	81.3 S.
21	25.5	5	4.155	0.9515	0.829	0.59	7.04	76.2 C.P.
21	"	5	4.1285	0.904	0.787	0.505	8.16	88.2 S.
20	36.5	5	4.316	0.9515	0.863	0.613	7.04	72.8 C.P.
20	"	5	4.248	0.968	0.818	0.600	7.08	73.1 S.

TABLE V.

No.	% Pb.	Pressure.	Weight.	Dia.	Length.	Volume.	Density.	% Solidity
23	16.5	5	4.200	0.9515	0.850	0.605	6.94	75.1 C.P.
23	"	5	4.160	0.905	0.834	0.536	7.75	83.8 S.
24	25.5	5	4.180	0.9515	0.820	0.5835	7.15	75.6 C.P.
24	"	5	4.132	0.965	0.800	0.585	7.06	74.7 S.
23	36.5	5	4.334	0.9515	0.863	0.614	7.05	72.0 C.P.
23	"	5	3.810	0.998	0.805	0.58	6.57	67.9 S.

TABLE VI.

No.	% Pb.	Pressure.	Weight.	Dia.	Length.	Volume.	Density.	% Solidity.
29	16.5	5	4.090	0.957	0.841	0.605	6.76	73.1 C.P.
29	"	5	4.050	0.918	0.812	0.537	7.55	81.6 S.
28	25.5	5	4.142	0.9515	0.826	0.587	7.05	74.5 C.P.
28	"	5	4.115	0.950	0.795	0.564	7.29	77.1 S.
26	36.5	5	4.328	0.9515	0.867	0.616	7.02	72.5 C.P.
26	"	5	4.249	0.982	0.843	0.631	6.73	69.5 S.

TABLE VII.

No.	% Pb.	Pressure.	Weight.	Dia.	Length.	Volume.	Density.	% Solidity.
37	16.5	5	4.030	0.953	0.837	0.5965	6.75	73.0 C.P.
37	"	5	4.000	0.935	0.824	0.566	7.06	76.3 S.
38	"	5	4.045	0.953	0.841	0.599	6.75	73.0 C.P.
38	"	5	4.041	0.935	0.828	0.568	7.12	77.0 S.
35	25.5	5	4.088	0.953	0.820	0.584	7.00	74.0 C.P.
35	"	5	4.083	0.913	0.787	0.515	7.93	83.9 S.
36	"	5	4.0955	0.953	0.826	0.588	6.96	73.6 C.P.
36	"	5	4.079	0.913	0.796	0.520	7.83	82.8 S.
33	36.5	5	4.2265	0.9535	0.854	0.609	6.94	71.7 C.P.
33	"	5	4.137	0.933	0.820	0.570	7.25	75.0 S.S.D.
34	"	5	4.2255	0.9535	0.854	0.608	6.96	72.0 C.P.
34	"	5	4.127	0.935	0.817	0.560	7.36	76.1 S.S.D.

obtained with comparatively low pressures. It will be noticed also that the three compositions do not vary substantially in their cold-pressing properties. These specimens were then sintered, using a 3 in. I.D. silica tube inserted in an electrically heated furnace. Before sintering, the furnace was at 1,000° C., and after inserting the tube 8 mins. was required before recovery to the sintering temperature, which in this case was 850° C. This temperature was held for 20 mins., and the tube extracted. The specimens cooled to room temperature in the tube in approximately 15 mins. An atmosphere of hydrogen was maintained in the tube. Before use it was dried by passing over calcium chloride and phosphorus pentoxide. The results after sintering are recorded in Table III. Considerable distortion took place with most of these specimens, with the exception of the 16.5% lead and the 25.5% lead under low pressures. Most of the 36.5% lead specimens were badly distorted, too much so for measurement and density determinations. The results, however, clearly indicated that maximum solidities are obtained at the lowest pressures. It was noticed that in most cases there was a slight loss in weight during sintering. This phenomenon is not entirely explained, but it is assumed to be partly due to the deoxidation and partly to loss of lead by volatilisation.

As it was found that the rapid rate of heating in these tests was responsible to a large extent for the distortion during sintering, further tests were made, employing a slower rate of heating to the sintering temperature. In this and subsequent work sintering was conducted in a tube 1½ in. I.D. Table IV records results made under 5 tons per sq. in., in which the heating time to 850° C. was 80 mins., and the sintering time 20 mins. at this temperature. Cooling down to room temperature took approximately 10 mins.

These results confirm the supposition that a slower heating to the sintering temperature reduces the amount of distortion. None of these specimens was distorted seriously. This experiment was again repeated, taking 1 hour to heat to the sintering temperature of 850° C., which was maintained for 30 mins. These results are reported in Table V, and they substantially conform to those recorded in Table IV. Still further tests were made by heating the specimens slowly to 350° C. over 30 mins. and then rapidly to 850° C. for another 20 mins. The sintering temperature was maintained for 30 mins. Results as shown in Table VI are again similar. From these and previous experiments it is clear that solidities in excess of 75% can be obtained without appreciable difficulty and undue distortion.

Work was continued with the intention of ascertaining optimum sintering temperatures in order to obtain maximum solidity without distortion. In all the subsequent pressings a pressure of 5 tons per sq. in. was employed. Results are reported in Tables VII to X. Table VII shows the solidities obtained by sintering at 650° C. for 20 mins. The time of heating to this temperature was 60 mins., and that of cooling to room temperature 10 mins. In each case recorded in these tables the upper line against the number of the specimens are particulars of the cold-pressed compact and the line immediately underneath are sintered compacts. It is interesting to note the considerable increase in density on sintering of the 36.5% lead alloy, compared with the minor changes in density of the other specimens. Table VIII records the results of sintering for 20 mins. at 750° C., time of heating up and cooling down being

TABLE VIII.

No.	% Pb.	Pressure.	Weight.	Dia.	Length.	Volume.	Density.	% Solidity.
40	16.5	5	4.079	0.951	0.845	0.600	6.79	73.4 C.P.
40	"	5	4.049	0.926	0.830	0.559	7.25	78.4 S.
41A	"	5	4.076	0.951	0.846	0.601	6.78	73.2 C.P.
41A	"	5	4.046	0.926	0.830	0.559	7.24	78.3 S.
38	25.5	5	4.155	0.951	0.831	0.590	7.04	74.5 C.P.
38	"	5	4.130	0.903	0.792	0.507	8.15	86.2 S.
39	"	5	4.164	0.951	0.822	0.581	7.12	75.4 C.P.
39	"	5	4.139	0.904	0.786	0.503	8.19	86.6 S.S.D.
36	36.5	5	4.223	0.952	0.852	0.606	6.97	72.0 C.P.
36	"	5	4.159	0.905	0.818	0.598	6.95	71.9 S.S.D.
37	"	5	4.220	0.952	0.854	0.607	6.96	72.0 C.P.
37	"	5	4.155	0.958	0.821	0.591	7.03	72.5 S.S.D.

TABLE IX.

No.	% Pb.	Pressure.	Weight.	Dia.	Length.	Volume.	Density.	% Solidity.
34	16.5	5	4.059	0.9545	0.850	0.605	6.71	72.5 C.P.
34	"	5	4.197	0.903	0.816	0.521	8.05	87.0 S.
35	"	5	4.070	0.9515	0.853	0.605	6.73	72.8 C.P.
35	"	5	4.207	0.900	0.818	0.52	8.09	87.5 S.
32	25.5	5	4.258	0.9515	0.860	0.611	6.96	73.6 C.P.
32	"	5	4.174	0.924	0.825	0.553	7.55	80.0 S.D.
33	"	5	4.348	0.956	0.866	0.621	7.00	74.0 C.P.
33	"	5	4.656	0.922	0.840	0.623	7.48	79.0 S.S.D.
30	36.5	5	4.120	0.957	0.800	0.574	7.18	74.2 C.P.
30	"	5	3.717	0.952	0.753	0.535	6.95	71.9 S.D.
31	"	5	4.173	0.952	0.835	0.595	7.01	72.5 C.P.
31	"	5	3.840	0.962	0.794	0.577	6.65	68.7 S.D.

* Gain in weight on pickup of lead.

† High loss of lead.

TABLE X.

No.	% Pb.	Pressure.	Weight.	Dia.	Length.	Volume.	Density.	% Solidity.
45	16.5	5	4.120	0.953	0.851	0.605	6.81	73.6 C.P.
45	"	5	4.086	0.908	0.818	0.528	7.74	83.7 S.
46	"	5	4.110	0.953	0.855	0.61	6.74	72.9 C.P.
46	"	5	4.081	0.908	0.821	0.531	7.67	82.9 S.
42	25.5	5	4.126	0.953	0.832	0.593	6.95	73.5 C.P.
43	"	5	4.110	0.941	0.806	0.561	7.32	77.5 S.D.
44	"	5	4.100	0.953	0.826	0.588	6.98	73.9 C.P.
41	"	5	4.050	0.958	0.796	0.55	7.36	77.9 S.D.
41	36.5	5	4.272	0.9535	0.859	0.612	6.98	72.4 C.P.
41	"	5	4.204	0.922	0.823	0.61	6.9	71.4 S.D.
42	"	5	4.277	0.9535	0.862	0.615	6.95	71.9 C.P.
42	"	5	4.210	0.977	0.822	0.616	6.84	70.7 S.D.

TABLE XI.

No.	% Pb.	Coining Pressure.	Weight.	Dia.	Length.	Volume.	Density.	% Solidity.
41A	16.5	10	4.046	0.954	0.658	0.47	8.61	93
43	"	10	4.086	0.954	0.653	0.466	8.76	94.7
46	"	10	4.081	0.954	0.647	0.463	8.81	95.3
40	"	As	4.049	0.926	0.830	0.559	7.25	78.4
40	"	Sintered	4.049	0.953	0.735	0.524	7.73	83.5
40	"	10	4.049	0.954	0.650	0.465	8.71	94.2
40	"	20	4.049	0.956	0.627	0.45	9.0	97.3
38	25.5	As	4.130	0.903	0.792	0.507	8.15	86.2
38	"	Sintered	4.130	0.953	0.677	0.482	8.56	90.6
38	"	10	4.130	0.954	0.630	0.45	9.17	97.0
38	"	20	4.130	0.955	0.617	0.442	9.34	98.8

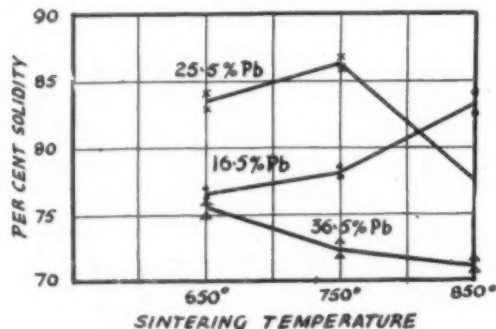


Fig. 4.—Solidities plotted against sintering temperatures.

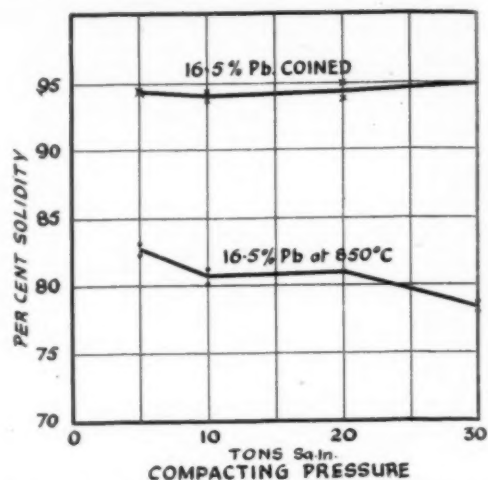


Fig. 5.—Solidities of 16.5% lead-alloy after compaction at various pressures.

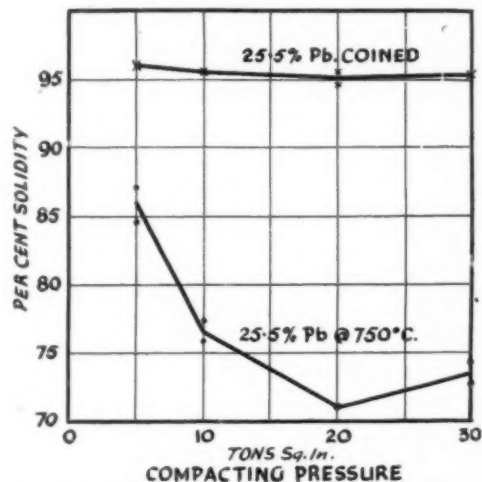


Fig. 6.—Solidities of 25.5% lead-alloy after compaction at various pressures.

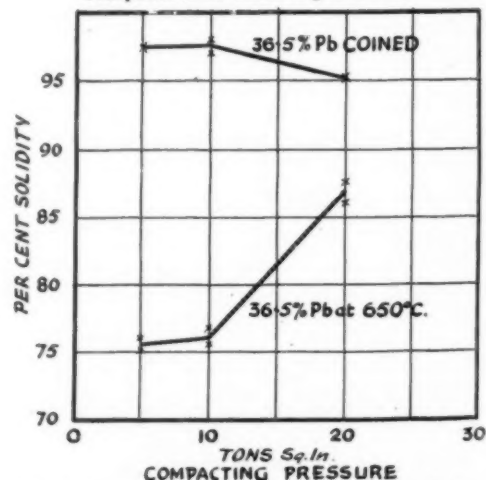


Fig. 7.—Solidities of 36.5% lead-alloy after compaction at various pressures.

TABLE XII.

No.	% Pb.	Pressure.	Weight.	Dia.	Length.	Volume.	Density.	% Solidity.
73	16.5	5	4.0345	0.953	0.835	0.596	6.75	73 C.P.
73	"	5	4.0020	0.909	0.805	0.523	7.65	82.7 S.
73	"	Specimen used for microsection.						
76	"	5	4.0220	0.953	0.831	0.593	6.78	72.1 C.P.
76	"	5	3.9890	0.910	0.799	0.520	7.67	83.0 S.
76	"	Coined	3.9890	0.954	0.639	0.457	8.73	94.4
77	"	10	4.0440	0.954	0.734	0.525	7.7	83.3 C.P.
77	"	10	4.0130	0.966	0.738	0.541	7.4	80.0 S.S.D.
77	"	Coined	4.0045	0.954	0.645	0.461	8.69	94.0
77	"	10	4.0280	0.954	0.730	0.522	7.7	83.3 C.P.
78	"	10	4.000	0.964	0.731	0.534	7.49	81.0 S.S.D.
78	"	Coined	4.000	0.954	0.641	0.458	8.74	94.5
79	"	20	4.0230	0.956	0.662	0.476	8.45	91.3 C.P.
79	"	20	3.9950	0.977	0.710	0.532	7.5	81.1 S.
79	"	Coined	3.9020	0.944	0.628	0.449	8.63	94.0
80	"	20	4.0110	0.956	0.658	0.473	8.47	91.5 C.P.
80	"	20	3.985	0.972	0.717	0.532	7.49	81.0 S.
80	"	Coined	3.9530	0.954	0.629	0.45	8.78	95.0
81	"	30	4.0115	0.956	0.636	0.456	8.79	95.1 C.P.
81	"	30	3.9800	0.970	0.746	0.551	7.23	78.1 S.D.
81	"	Coined	3.9765	0.954	0.632	0.453	8.78	94.9
82	"	30	3.9520	0.956	0.630	0.453	8.72	94.3 C.P.
82	"	30	3.9160	0.968	0.733	0.539	7.26	78.5 S.D.
82	"	Coined	3.8920	0.954	0.623	0.445	8.74	94.5

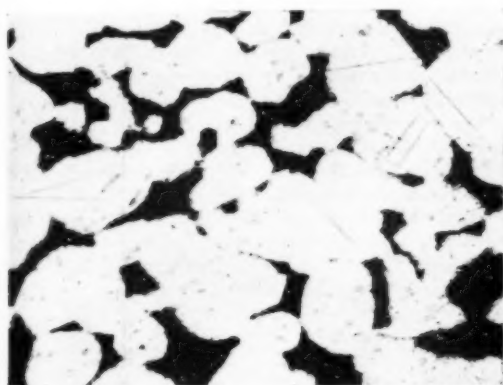


Fig. 8.—16.5% lead alloy, specimen No. 76, sintered at 850° C. Section parallel to direction of pressure in die and unetched. $\times 500$.

similar. Table IX shows the results of specimens sintered to 850° C., in which the heating up occupied 70 mins. to sintering temperature, the specimens being extracted immediately the sintering temperature was obtained. Table X shows various results on sintering at 850° C., in which the time of heating to the sintering temperature was 60 mins. and the sintering itself occupied 20 mins. All the specimens sintered at 950° C. exuded lead so badly that it was not possible to make accurate density observations, and comparing these results it is observed that increasing temperature affects the percentage solidity concerned in the case of the 16.5% lead alloy, but it does not make any substantial change in the case of either the 25.5% lead alloy or the 36.5% lead alloy. From these results it is assumed that of the four temperatures concerned the optimum temperatures for the three compositions, 16.5%, 25.5%, and 36.5% lead are 850°, 750°, and 650° C. respectively. It will be appreciated, however, that this work would have to be repeated at much narrower temperature intervals before any precise optimum sintering temperature can be selected. Per cent. solidities plotted against sintering temperatures are shown in Fig. 4.

In order to improve the percentage solidity of the sintered specimens they were repressed again in the same die under various pressures. This after-treatment is

TABLE XIII.

No.	% Pb.	Pressure.	Weight.	Dia.	Length.	Volume.	Density.	% Solidity.
67	25.5	5	4.0530	0.9535	0.815	0.582	6.96	73.7 C.P.
67	"	5	4.000	0.906	0.776	0.500	8.00	84.6 S.
67	"	Specimen used for microsection.						
68	"	5	4.0240	0.9535	0.806	0.575	7.00	74.1 C.P.
68	"	5	4.000	0.899	0.767	0.486	8.23	87.0 S.
68	"	Coined	3.9995	0.953	0.610	0.435	9.19	97.2
69	"	10	4.0410	0.954	0.697	0.499	8.1	77.5 S.D.
69	"	10	4.0165	0.990	0.699	0.548	7.33	85.7 C.P.
69	"	Coined	3.6780	0.954	0.563	0.403	9.13	96.5
70	"	10	4.042	0.954	0.695	0.496	8.15	86.3 C.P.
70	"	10	4.019	1.008	0.700	0.559	7.18	76.0 S.D.
70	"	Coined	3.9000	0.954	0.599	0.428	9.12	96.5
71	"	20	4.023	0.955	0.633	0.454	8.75	92.5 C.P.
71	"	20	4.0070	0.988	0.779	0.597	6.7	71.0 S.
71	"	Coined	3.9200	0.954	0.603	0.434	9.1	96.3
72	"	20	4.0620	0.955	0.640	0.459	8.84	93.5 C.P.
72	"	20	4.045	—	—	—	—	S.D.
72	"	Coined	3.5995	0.954	0.555	0.397	9.06	95.8
73	"	30	4.0340	0.961	0.615	0.446	9.04	95.7 C.P.
73	"	30	4.0150	0.977	0.763	0.573	7.00	74.1 S.D.
73	"	Coined	3.965	0.954	0.611	0.436	9.1	96.3
74	"	30	4.024	0.961	0.609	0.442	9.1	96.3 C.P.
74	"	30	4.0150	0.978	0.779	0.585	6.85	72.5 S.D.
74	"	Coined	3.949	0.954	0.606	0.433	9.13	96.5

TABLE XIV.

No.	% Pb.	Pressure.	Weight.	Dia.	Length.	Volume.	Density.	% Solidity.
59	36.5	5	4.0560	0.954	0.814	0.581	6.98	72.1 C.P.
59	"	5	4.1180	0.947	0.788	0.555	7.41	76.5 S.D.
59	"	Coined	4.019	0.952	0.595	0.424	9.46	97.8
60	"	5	4.0200	0.954	0.806	0.576	6.99	72.2 C.P.
60	"	5	3.9590	0.947	0.775	0.545	7.27	75.2 S.D.
60	"	Specimen used for microsection.						
61	"	10	4.0260	0.955	0.718	0.514	7.83	81.0 C.P.
61	"	10	3.966	0.982	0.715	0.541	7.34	75.8 S.
61	"	Coined	3.9365	0.9535	0.580	0.415	9.48	98.0
62	"	10	4.0300	0.955	0.705	0.505	7.98	82.5 C.P.
62	"	10	3.9710	0.981	0.706	0.534	7.44	76.9 S.
62	"	Coined	3.8875	0.954	0.577	0.413	9.42	97.5
63	"	20	4.0300	0.956	0.645	0.4635	8.69	89.9 C.P.
63	"	20	3.9740	0.958	0.650	0.469	8.47	87.6 S.
63	"	Coined	3.9715	0.953	0.603	0.43	9.23	95.4
64	"	20	4.045	0.956	0.649	0.466	8.68	89.6 C.P.
64	"	20	4.016	0.961	0.656	0.476	8.43	87.1 S.
64	"	Coined	3.999	0.954	0.606	0.434	9.21	95.3
65	"	30	4.035	0.957	0.624	0.45	8.96	92.5 C.P.
65	"	Specimen sweated badly.						
66	"	30	4.0275	0.957	0.625	0.45	8.91	92.4 C.P.
66	"	Specimen sweated badly.						

C.P.—Cold Pressed.

S.—Sintered.

S.D.—Slightly Distorted.

D.—Distorted.

subsequently referred to as coining. Table XI shows the densities and percentage solidities obtained in 16.5% and 25.5% lead alloys. It will be observed that considerable increase in solidities was obtained with comparatively low coining pressures. With a pressure of 20 tons per sq. in. a comparatively normal industrial pressure, a solidity approaching 100% is recorded.

It was now felt desirable to repeat some of this work, using a series of varying pressures at those sintering temperatures which are most suitable for the particular composition concerned. Table XII shows the results of specimens pressed under 5, 10, 20 and 30 tons per sq. in. On the 16.5% lead alloy the sintering took place for 20 mins. at 850° C., the time of heating to the sintering temperature being 60 mins. and the time of cooling 10 mins. In this table the first line against the specimen number records the cold-pressing results, the second line the sintering results, and the third line the results after coining under 10 tons per sq. in. These results are shown also in Fig. 5. Similar results for the 25.5% lead alloy sintered at 750° C. are shown in Table XIII and Fig. 6, and for the 36.5% lead alloy, sintered at 650° C. in Table XIV and Fig. 7. In some cases the compacts, especially the 36.5% lead alloy specimens, show distortion, and one or two cases of exudation of lead was observed. In almost every case there is a slight loss of weight on sintering, and there are some examples of high losses. In one or two cases increases in weight during

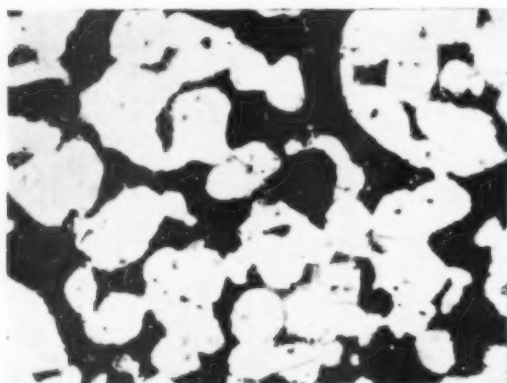


Fig. 9.—25.56% lead alloy, specimen No. 67, sintered at 750° C. Cross section, unetched. $\times 500$.

sintering also occurred, and these are believed to be due to pick-up of lead from an adjacent specimen by volatilization. This work confirms that the highest percentage solidities are obtainable with the lowest pressing pressures, but with one apparent exception in the case of the 36.5% lead alloy under 20 tons per sq. in. The graphs also show that the per cent. solidities after coining are practically independent of previous treatment.

Conclusions

The powders examined have extremely good cold-pressing properties, and are quite remarkable in the fact that percentage solidities in excess of 80% are obtainable without addition of lubricants with compact-

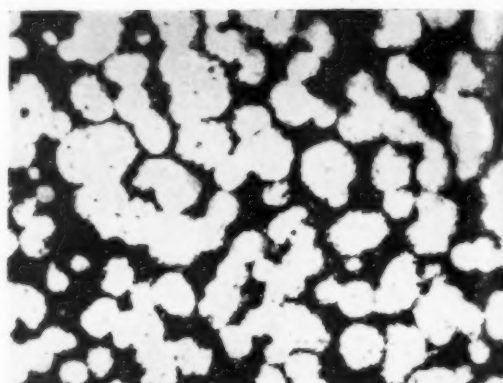


Fig. 10.—36.3% lead alloy, specimen No. 60, sintered at 650° C. Cross section, unetched. $\times 500$.

ing pressures as low as 5 tons per sq. in. It is quite possible to obtain bearing materials using these powders employing pressures of the order of 5 tons per sq. in., sintering temperatures over the range of 650° to 850° C., and coining pressures of 10 tons per sq. in. and securing a practically non-porous product. Photomicrographs of the three compositions (Specimen Nos. 60, 76 and 67) are attached (Figs. 8, 9 and 10).

This work indicates also the treatments desirable in order to obtain porous alloys which, of course, could be oil-impregnated if desired. Similar if not more successful results are also obtainable with copper-tin-lead alloy powders. It might be mentioned that no difficulty is experienced in bonding these alloys by conventional methods to a steel backing.

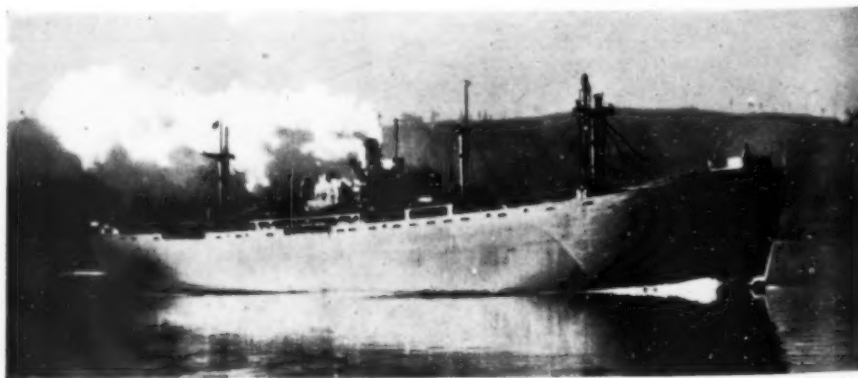
Welded Ship Withstands Severe Damage A Compliment to Welders

EVIDENCE of the stability of welded structures is accumulating, especially in its application to vessels in merchant service. A recent example is that of the *Edgar Allan Poe*, an all-welded cargo vessel shown in the accompanying illustration. This vessel was struck by an enemy torpedo amidship and the manner in which she stood up to the blow caused Mr. E. S. Land, Chairman of the United States Maritime Commission, to send congratulations to the workers of the Oregon Shipbuilding Corporation.

This vessel was struck amidships by an enemy torpedo, but her crew stood to her guns and continued to blaze away until the Jap submarine was sunk. The entire engine-room of the *Edgar Allan Poe* was damaged; the steam and fuel pipes ruptured; the lower decks lifted, and great damage done throughout the midship structures. But the bulkheads held. No water reached any

cargo space. The vessel was later towed to port with its entire cargo intact. Two seamen gave their lives, so that this ship, with its vital war material, should reach the Allied fighting forces in the South Pacific.

As Mr. Land said in his telegram: "The performance of the *Edgar Allan Poe* is a credit to every worker of the Oregon Shipbuilding Corporation, and I know you will take pride in its accomplishment."



The *Edgar Allan Poe* on a trial run.

Courtesy of Lincoln Electric Co., Ltd.

The Analysis of English Fluorspars and their Associated Minerals

By G. H. Osborn,

International Alloys, Ltd.

The increasing use of home-mined fluorspars for metallurgical practice, and in many other industries, has imposed a strain on English mines, only a few of which have flotation and washing plants to obtain the necessary grades. Consequently the material is often heavily contaminated with associated minerals. The task of analysing such such material is not easy, and in addition an analysis of the ions present gives little insight of the true mineralogical composition. In this article the author submits a scheme by which the chemist may obtain results approximating to the truth.

AS is well known, fluorspar or fluorite occurs abundantly in England in Derbyshire and Durham, where it is associated with mineral veins, and where it was long mined for specimens of gem quality and decorative purposes. The increasing use of this material as a flux in metallurgical practice, and its use in many other industries, has led to the revival of mining in these two famous localities. The outbreak of war, with the consequent cutting off of foreign supplies, coincident with the rising demand, has led to a great increase in output, and this, in turn, has caused a great strain on the English mines. Unfortunately, only a few of the larger concerns have flotation plants and the washing plants necessary to obtain the 98-99% grades. Thus, the large bulk of the material is supplied by small mining concerns and is often heavily contaminated with associated minerals partly crushed together. The works chemist, often quite ignorant of mineralogy, is confronted with the task of analysing an intimate mixture of some 12 or 15 minerals. A typical chemical analysis is only concerned with the ions present, and has often no relation to their true distribution as natural minerals. It is therefore little wonder that often only two or three radicles are chosen for analysis, and the material judged on this finding. The lack of literature dealing with such complicated analysis is partly responsible for this, and as the author has seen very many erroneous analyses made and accepted in good faith, this paper is an attempt to lay out a scheme whereby the chemist without much, or even any, knowledge of mineralogy may obtain results approximating to the truth, and it is to be hoped also that it may prove of use and interest also from a chemical aspect to such mineralogists who undertake their own analyses.

The minerals mainly associated with such spars and nearly always found by us to be present are:—

	Usual Percentages.
(a) Quartz.....	(SiO ₂)..... 0.5 — 8.0%
(b) Calcite.....	(CaCO ₃)..... 2.0 — 15.0%
(c) Dolomite.....	(CaCO ₃ , MgCO ₃)..... 0.5 — 2.0%
(d) Witherite (rare).....	(BaCO ₃)..... Tr. — 0.5%
(e) Rhodochrosite.....	(MnCO ₃)..... Nil — 0.06%
(f) Galzbyite.....	(FeCO ₃)..... 0.05 — 0.3%
(g) Smithsonite.....	(ZnCO ₃)..... 0.05 — 2.0%
(h) Cerussite.....	(PbCO ₃)..... Nil — 0.06%
(i) Lindeite.....	(ZnS)..... 0.05 — 0.5%
(j) Gypsum.....	(PbS)..... 0.1 — 2.0%
(k) Pyrite.....	(BaSO ₄)..... 2.0 — 10.0%
(l) Gypsum.....	(CaSO ₄ , 2H ₂ O)..... 0.5 — 5.0%
(m) Hematite and Limonite.....	(Fe ₂ O ₃)..... 0.5 — 5.0%
(n) Malachite (rare).....	(CuCO ₃ , Cu(OH) ₂)..... Nil — 0.05%
(o) MnO ₂ + MnO ₂ Hydrates (Wad).....	0.05 — 0.5%
(p) Clay.....	2.0 — 10.0%

Other minerals are sometimes encountered and should be looked for, but they are seldom present in amounts which would seriously interfere with the calculation. Titanium minerals have very seldom been encountered, though the ores are frequently tested for this element. Pyrite, Marcasite and Chalcopyrite should be looked for as they may be present, though the author has never encountered them.

Sampling

The material is usually delivered in truck loads, and consists of fairly small spar, some 10% of which would not pass a $\frac{1}{4}$ -in. sieve, and the remainder would pass a $\frac{1}{4}$ -in. sieve with a small percentage of very fine material. The truck is sampled by an experienced sampler, who collects a sample of about 5,000 grms. This is halved and quartered in the usual manner in the laboratory, and dried to constant weight at 105°–110° C. in a constant drying oven. Great care must be taken not to exceed this temperature, since at 120° C. Gypsum (CaSO₄·2H₂O) is converted to the hemihydrate, and an erroneous moisture and Gypsum content would be reported.

Clay

Clay is often found up to 10%, and seldom less than 2%. It is therefore important to separate it and estimate it carefully, since if not removed its presence complicates the analysis of the minerals to an almost impossible extent. 500 grms. of the dried material are weighed out on an accurate scale and then placed in a very large beaker (2–3 litre). This is then filled with boiling water, and the whole mass strongly agitated with a glass rod. The fine suspension is then decanted rapidly through a 300-mesh sieve, which permits the passage of the suspended clay but retains any mineral particles. This operation is repeated until such time as no further suspensions occur upon the addition of fresh water; the material is then dried (105°–110° C.) and reweighed, and the percentage of clay on the dried material estimated. It was found necessary to use the 300-mesh, since larger meshes permitted fine particles of fluorspar to pass through; the clays passing the 300-mesh are free of particles of fluorspar, but will contain a small but negligible percentage of calcium fluoride dissolved by the water.*

* This is, of course, only true of partly crushed fluorspar where particles are fairly large. In finely ground fluorspar it will be necessary to collect and dry the clay and determine the fluorspar in it.

Physical Examination

At this stage when the material is clean and dry a physical examination should be made from which much valuable information may be gained. A microscope is very valuable, but in general an experienced eye may obtain much valuable information with the aid of a hand-lens. Some of the larger lumps may be identified at once, but usually sorting out tests are required. Passing the material over the electromagnets is not likely to remove much except the hematite, but subsequent treatment with heavy liquids will give a very good idea of the minerals present and the amounts to be expected. The first treatment with bromoform (2.9) sp. gr. will separate out the Quartz (2.6), Calcite (2.7), Gypsum (2.3), and further treatment with methylene iodide (3.33) will separate the Fluorite (3.1) from the Rhodochrosite (3.5), Witherite (4.3), Barite (4.5), Galena (7.4), Blende (4.0), Chalybite (3.83), Malachite (3.9), Smithsonite (4.3), Cerussite (6.46). A still further treatment with Clerici solution (4.1) will separate out the very heavy minerals. Any coloured minerals present, e.g., Galena, Malachite, Pyromorphite (found very rarely), Rhodochrosite, Wad, etc., may be hand-picked and identified by qualitative tests. Streak tests may also be used with advantage, e.g., differentiation between Hematite (red) and Limonite (yellow-brown).

The washed and dried material is then halved and quartered, and a 50-grm. portion crushed and ground in an agate mortar until the whole passes the 100-mesh sieve.

The Analysis Proper

Most methods found in textbooks (1, 2, 3, 5, 6.) commence by attacking with 20% acetic acid and estimating the calcium and magnesium in the filtrate and recording this as Calcite and Magnesite, but this has been found to be quite erroneous since some fluorite is always soluble. The amount, however, is never constant, varying with the amount and type of other constituents present, and cannot, therefore, be assumed. As the result of many tests the solubility was found to vary between 1.5 and 5%. In addition it was found that under these conditions 80-85% of the gypsum present is soluble, and since it is often present in these mixtures up to 5%, it is easy to see the errors arising for the unwary.

It was found absolutely necessary to estimate the carbon dioxide evolved, and to calculate the soluble fluoride.

Calcite, Magnesite, Witherite, Cerussite, Smithsonite, Chalybite, Rhodochrosite and Gypsum.—5 grms. of the powder are weighed in a CO₂ apparatus of the "Schrotter" type, and after 200 ml. of 40% acetic acid has been added the whole is heated to expel the CO₂ which is trapped in soda-lime, precautions for the absorption of moisture having been taken. When the reaction has ceased the CO₂ is estimated both by loss in the apparatus and as a check by weight in the soda-lime tube. The

solution is then filtered, washed well, and made up to 500 ml.; the residue is reserved. The filtrate contains the acetates of calcium, barium, lead, iron, manganese, copper, most of the gypsum and some fluorite. A 100 ml. portion is brought to the boil, 5 ml. of concentrated hydrochloric acid added, and 10 ml. of barium chloride 10% solution added. The precipitated barium sulphate is estimated in the normal manner and calculated to CaSO₄·2H₂O. A 200 ml. portion is raised to boiling point, 5 ml. hydrochloric acid added, followed by 4 ml. of 50% sulphuric acid. This will precipitate any barium present, as the sulphate, whence it may be estimated and calculated to BaCO₃. If it is not present, or after it has been removed, hydrogen sulphide is passed through the solution for 10 mins., and the lead and copper sulphides, if present, are filtered off, redissolved in nitric acid, and after suitable dilution electrolysed; copper will deposit on the cathode as the metal, and lead on the anode as the peroxide. Both are weighed and calculated to their carbonates. The filtrate is boiled to remove the hydrogen sulphide, a slight excess of ammonia added, followed by 10 ml. of bromine water, and the solution boiled for 5 mins.; this will precipitate the iron and manganese together and they are filtered off (reserve filtrate), redissolved in hydrochloric acid, and the solution boiled strongly; 5 grms. of ammonium chloride are added and the iron alone reprecipitated by ammonia. If there is much iron present it may be burnt off and weighed as the oxide, but it is more often dissolved in 5 ml. of hydrochloric acid plus 5 ml. of nitric acid, the whole boiled down to about 4 ml., diluted to 100 ml. and 25 ml. of 10% potassium thiocyanate added, and the iron estimated colorimetrically against known standards and calculated to the carbonate. The manganese is reprecipitated in the filtrate by boiling after the addition of bromine water, is filtered off and dissolved in 5 ml. nitric acid and 5 ml. sulphuric acid, diluted to 100 ml., and after the addition of 10 ml. of 25% ammonium-persulphate solution, plus 10 ml. of a 0.3%, silver-nitrate solution, the manganese is estimated colorimetrically or titrated against standard sodium-arsenite solution when cold. The manganese thus found is calculated to the carbonate.

The reserved filtrate is now gassed with hydrogen sulphide, and the zinc sulphide filtered off, dried and burnt off to the oxide and calculated to the carbonate. The filtrate is acidified with nitric acid, the excess hydrogen sulphide boiled off and an excess of ammonia added; the calcium is precipitated as the oxalate, and estimated either as the oxide or volumetrically against potassium permanganate. In the filtrate magnesium is precipitated by oxine, and after drying at 105° C. and weighing it is calculated to the carbonate, and then to Dolomite CaCO₃·MgCO₃, after subtracting the necessary CaCO₃ from the calcite found, since magnesite, as such, is not found in England.

Calculations

The percentage of ZnCO₃, PbCO₃, FeCO₃, BaCO₃, MgCO₃ and CuCO₃·Cu(OH)₂ are known, and also the total CO₂; any CO₂ not accounted for is therefore calculated to the calcium as CaCO₃. The calcium known to be present as the sulphate and the calcium present as the carbonate are subtracted from the total calcium found, and the remaining calcium calculated to the fluoride.

1 *The Technical Analysis of Ores and Metallurgical Products*, p. 46. F. G. Hills, 1937.

2 Landell and Hofman, *Bureau of Standards Journal of Research*, April, 1929, p. 674.

3 Treadwell and Hall, Vol. 2, 9th ed., p. 398.

4 Thorpe's *Dictionary of Applied Chemistry*, 4th ed., vol. v, 1941, p. 283.

5 V. I. Lisitsyn and S. T. Volkov, *Zavodskaya Lab.*, 8, 943-7 (1939); *Atom Refract. Khim.*, 1940, No. 329-30.

6 Scott's *Standard Methods of Chemical Analysis*, 5th ed., vol. 1, p. 419, 1939.

Galena.—5 grms. of the powdered material are digested in a beaker with 100 ml. of 10% nitric acid for half an hour on a hot-plate, and then filtered off. The filtrate is diluted to 250 ml. and then electrolysed at 2.5 amps. and 1,000 r.p.m. for half an hour, using a platinum anode and cathode with a glass stirrer. Any lead present is deposited as the peroxide on the anode, and copper on the cathode as the metal. After drying and weighing the lead known from the previous experiment to be present as the carbonate is deducted, and the remainder calculated to the sulphide. The copper may be weighed and checked against the copper found previously; they should be in agreement as no other copper minerals have been so far encountered.

Blende.—2 grms. are digested in a beaker on a hot-plate with 100 ml. of 5% sulphuric acid for 15 mins., and are then filtered off. The filtrate is made alkaline by adding caustic soda or potash, and is then boiled. Any deposit of bases, etc., are filtered off and the filtrate diluted to 250 ml.† This solution is then electrolysed for half an hour at 2 amps. and 1,000 r.p.m., using a copper-coated cathode and a glass stirrer. The zinc is deposited as the metal and dried and weighed as such; any zinc known from the previous experiment to be present as the carbonate is deducted and the remainder calculated to the sulphide.

Hamatite.—1 gm. of the material is refluxed for 1 hour with 20 ml. of concentrated hydrochloric acid. This treatment takes all the iron into solution, and after filtration the iron is estimated by reduction with stannous chloride and titration with N/10 potassium permanganate, or by any other reliable method such as the dichromate-titanous chloride, etc. When the total iron has been found, that known to have been present as the carbonate is subtracted and the remainder calculated to the oxide.

MnO₂ + MnO₂ Hydrates.—1 gm. of the material is placed in a platinum crucible, 10 ml. of concentrated sulphuric acid, and then evaporated to dryness. When cold it is fused with potassium bisulphate and then extracted in a 100 ml. of water containing 5 ml. nitric acid and 5 ml. sulphuric acid. The temperature of the solution is raised to 80° C., 10 ml. of a 0.3% solution of silver nitrate added, followed by 10 ml. of 25% ammonium-persulphate solution. When the colour has fully developed the solution is cooled and titrated against standard sodium-arsenite solution. From the total manganese found, that known to be present as the carbonate is deducted and the remainder calculated to the dioxide.

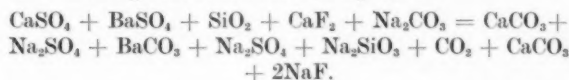
Quartz.—If the clay has been removed carefully the only form of silica usually encountered is quartz. Most of the methods proposed for its evaluation have been investigated, and the following method has been found to be most simple and to give very reliable results, both on synthetic mixtures and ores provided that carbonates, etc., have been previously removed by the acetic-acid treatment.

The residue from the 5 grms. treated with acetic acid is dried and burnt off in a platinum crucible and weighed, 10 ml. of (A.R.) hydrofluoric acid are then added and the whole evaporated, very slowly to avoid spitting, on

a hot-plate. When quite dry the crucible is heated strongly to remove the last traces of hydrofluoric acid and then cooled and reweighed, and the loss in weight calculated to SiO₂. For accurate work a second evaporation is advisable to ensure complete removal of silica.

Barite, Gypsum and Fluorite.—The residue after the removal of the silica consists of calcium fluoride, any barium and calcium sulphates which may be present, the zinc and lead sulphides, and iron oxides. To this residue is added 1 to 2 grms. of pure silica, without which the fluorite will not fuse completely, and 10 grams of fusion mixture and the mixture well fused. Care should be taken that nothing is lost, as the evolution of carbon dioxide is fairly rapid. The melt is extracted in 400 ml. of boiling water and the insoluble part filtered off.

If barium and calcium sulphates are both present then the following reactions theoretically occur:—



Thus the carbonates should be insoluble, and the sodium salts should be soluble. Though this may be found stated in print (3, 7), in point of fact experiments show that two-thirds of the sodium fluoride is occluded with the precipitated carbonates, and the recovery must be made from both portions.

The filtrate (A) is acidified with hydrochloric acid and adjusted to 500 ml. A 100-ml. portion of this is taken and 200 ml. of water added, and the solution brought to the boil. 15–20 ml. of 10% barium-chloride solution is added and the sulphates precipitated and estimated in the usual manner, but taking care to wash with ammonium acetate to remove any lead sulphate. This will give the total sulphates.

The residue of insoluble carbonates is digested in 100 ml. of 20% hydrochloric acid and diluted to 500 ml. (B). A 100-ml. portion is taken, diluted to 300 ml., and 5 ml. of 1/1 sulphuric acid added. This will precipitate any barium as the sulphate, but not the calcium, owing to the greater solubility of the calcium salt (0.1619¹⁰⁰). The precaution of washing with ammonium acetate should be taken here also. The barium sulphate thus found subtracted from the total sulphates $\times 0.737$ will give the gypsum. To the gypsum thus found is added the gypsum known to be soluble in the acetic acid, and the total gypsum thus obtained. 50 ml. of both (A) and (B) are combined and the sulphates removed by the addition of barium chloride and subsequent filtration; this is necessary to avoid precipitation of calcium sulphate at a later stage. To the filtrate is added a few grammes of tartaric acid to keep the iron in solution, and the solution brought to the boiling point. 20 ml. of 10% calcium chloride solution are now added, followed by a slight excess of ammonia. This will precipitate all the calcium fluoride plus the silica. After standing for 1 hour the precipitate is filtered off, washed well, dried and burnt off in a platinum crucible. When cool 10 ml. of hydrofluoric acid are added and evaporated on the hot-plate to dryness. The silica will be removed and the calcium fluoride may be weighed and estimated. The calcium

† If a large quantity of bases are present it is advisable to redissolve them in sulphuric and reprecipitate them to avoid occlusion of the zinc. On no account should nitric acid be used.

fluoride may be contaminated with iron as the fluoride (FeF_3), and in this case, after the weight has been found, the residue is refluxed with hydrochloric acid, as mentioned under hæmatite, the iron found, calculated to the fluoride and subtracted from the total, this leaving the pure calcium fluoride. To the amount found is added the amount known to be soluble in acetic acid, and the total fluorite thus obtained.

Results

Using these methods upon synthetic mixtures made from pure ores the following results were obtained:—

	Taken.	Found.
(a) Fluorite.....	60%	59.87%
Barytes.....	20%	19.86%
Gypsum.....	10%	10.15%
Calcite.....	5%	5.03%
Quartz.....	5%	4.92%
(b) Fluorite.....	50%	49.88%
Barytes.....	20%	19.90%
Gypsum.....	5%	5.13%
Quartz.....	10%	10.05%
Galena.....	1.0%	1.06%
Blende.....	1.0%	1.04%
Calcite.....	10.0%	10.15%
Magnesite.....	3.0%	3.07%

A typical analysis of a heavily contaminated Derbyshire spar is also given.

H_2O at 110°C . 5.6%.

DRIED MATERIAL.

Clay.....	7.3%	Cerussite.....	0.08%
Calcite.....	8.5%	Galena.....	1.56%
Hæmatite.....	2.6%	Calamine.....	0.97%
Chalybite.....	0.2%	Blende.....	0.16%
Rhodochrosite.....	0.07%	Witherite.....	Trace.
$\text{MnO}_2 + \text{MnO}_2$ Hydrates.....	0.12%	Barytes.....	6.9%
Malachite.....	0.04%	Gypsum.....	2.6%
Dolomite.....	0.44%	Quartz.....	7.6%
		Fluorite.....	60.91%

I wish to thank the directors of Messrs. International Alloys, in whose laboratories these investigations were undertaken, for permission to publish this paper; also Messrs. F. Gilbert, J. Clark and C. Jukes, of this Company, for valuable suggestions and assistance with the experimental work.

Addendum

Since the above paper has been written it has been kindly pointed out to the author, by Dr. L. J. Spencer, F.R.S., and also by Mr. L. C. Chadwick, M.A., of the Mineral Resources Department, Imperial Institute, that minerals of the Ankerite group, such as "Brown Spar," which are isomorphous mixtures (Ca , Mg , Fe , Mn) CO_3 , and which do not give off their CO_2 in acetic acid, are sometimes encountered, especially with Weardale and Alston fluorites. If these are observed during the physical examination they should be removed and estimated separately to avoid interference with the proposed scheme.

Cerium and some of its Applications

By Leo David, M.Inst.M.*

Cerium, one of the rare earth metals, has powerful reducing properties and exerts a refining action on other metals and alloys. In the form of cerium standard alloy its best known use is in the production of flints for spark-emitting instruments, particulars of which are given in this article.

CERIU belongs to the group of elements known as "Rare Earths Metals," and is contained in particular in monazite.* A typical analysis of India monazite sand is as follows:—

ThO_2	8.49	TiO_2	0.50
CeO_2	28.27	Al_2O_3	0.12
La_2O_3	30.17*	CaO	0.65
Fe_2O_3	0.12	P_2O_5	2.7.72
Y_2O_3	0.70	SiO_2	0.72

* Combined lanthanum, praseodymium, neodymium, and samarium oxides.

Monazite is produced for the extraction of thorium oxide, which is used in the gas mantle and electric industry. It is possible to extract rare earths metals individually from the rare earth oxides remaining as residues after the thorium-oxide has been extracted, but no commercial application has been found for the individual rare earths metals. However, the combined rare earths metals have found employment for different purposes. The alloys containing the different rare earths metals are known under the names of "cerium alloy," "Misch metals," "Cer-metals," "mixed metals," "cerite alloy." The following is a typical analysis of this alloy, which, for matter of simplicity, is called hereafter "cerium standard alloy":—

Cerium.....	51—53	Lanthanum.....	22—25
Neodymium.....	15—17	Samarium.....	2—3
Praseodymium.....	3—4	Terbium plus	
Calcium.....	Traces	Yttrium.....	3—3
(approx. 0.014)		Calcium carbide.....	(calculated)
Carbon.....	Traces	Iron.....	5
(approx. 0.054)		Silicon.....	Traces
Aluminium.....	Traces		

* See METALLURGIA, 28, 1943, pp. 71-72 for further information about this mineral.

Apart from this standard cerium alloy, relatively pure cerium metal containing 95% Ce, balance iron, ferro-cerium 70% Ce, 30% Fe, lanthanum metal and thorium metal are also available in commercial quantities.

The best-known use for the cerium standard alloy is in the production of flints for cigarette lighters, gas lighters and other spark-emitting instruments, because all rare earths metals, with the exception of lanthanum, if alloyed with a sufficient percentage of iron, will produce sparks when struck or scratched with iron. The iron is usually added to the cerium standard alloy at the rate of 5 to 40% to the molten alloy in a graphite crucible under cover of melted sodium chloride or barium chloride. Some flint manufacturers add boron to increase hardness, air resistance, and combustion heat; whilst other manufacturers add 4-7% silicon or 5-7% calcium. Sometimes tin, lead, zinc or cadmium is added, and it may be said that practically every flint manufacturer has developed a special alloy and a special method of production. The spark-emitting property of the rare earths metals is due to their low-ignition temperature. (Cerium approximately 150°C .) Fragments of the metal, struck off by means of wheels in the case of lighter flints, are raised to ignition temperature by the heat of friction. It may also be that the formation of sub-oxide on the surface of the metal is either wholly or partially responsible for the emission of sparks. Experimental heating of rare earths metals in a sealed glass bottle produced black powder on the surface of the metal, and on opening the bottle this powder ignited immediately. According to

an Austrian patent, flints are manufactured by pressing a powdered rare earths metal alloy, the pressed parts are heated slowly to red heat and the sintered product is said to contain a large amount of sub-oxide, to be air-resistant, and highly spark-emitting.

The well-known flints are usually made from pressed sticks or rods. Their production is a very difficult operation requiring expensive and delicate equipment; approximately 3,100 flints, 5 mm. in length, and in the most frequent diameter of 24 mm., will weigh only 1 lb.

Cerium standard alloy will alloy readily with aluminium, iron, copper, magnesium, zinc and bismuth. The alloy dissolves easily in diluted mineral acids, and will ignite when heated in air or oxygen and burns with great brilliance. The alloy also amalgamates with mercury. For pyrotechnical purposes cerium amalgamate, with approximately 40% cerium, was produced by heating particles of cerium in mercury vapour to 500°-600° C. under vacuum, and this amalgam is said to ignite on exposure to air. The inventor's idea was to use this amalgam for tracer bullets and similar devices.

Less known is the use of cerium standard alloy in the iron and steel industry. In consequence of the high heat of combustion these metals have powerful reducing properties. Their affinity for oxygen is very great, and similar to aluminium and magnesium. The following indications will be of interest:—

Element,	Melting Point, °C.	Heat of Formation of Oxide-kilogram-calories per gramme Formula Weight.
Cerium (Ce)	645	Ce, 20 234.9
Lanthanum (La)	826	2La, 30 456.9
Neodymium (Nd)	840	2Nd, 30 435.1
Praseodymium (Pr)	940	Pr, 20 215.1
Samarium (Sm)	1,300	
Terbium (Tb)	—	
Yttrium (Y)	1,490	

In iron and steel alloys the cerium standard alloy will exert refining action by combining the oxygen, sulphur, nitrogen and phosphorus and transferring them to slag. This action is greatly improved by employing cerium standard alloy in association with one or more metals having a similar action on the impurities, such as aluminium and calcium in particular. The following alloys have been suggested as being especially suitable for this purpose:—

25-60% vanadium, balance iron.	5-10% cerium,
25-60% aluminium, balance iron.	5-15% cerium,
5-15% silicon, balance iron.	5-15% cerium,
5-15% calcium and/or 5% magnesium, balance iron.	5-15% cerium,
5-30% titanium, balance iron.	5-15% cerium,

In the case of the vanadium-containing alloy, it has been suggested that this alloy be introduced in powder form, or in the form of small pieces, into a steel bath; the high activity of the cerium will cause the alloy to act as a medium for combining the bulk of the oxygen, nitrogen, sulphur, etc., dissolved in the bath and transfer these to the slag.

The use of cerium offers the advantage that the portion of vanadium which otherwise would have reacted with the injurious substances is saved, as its place in the reaction is taken by the cerium. Also, as a consequence it is easier to adjust the amount of vanadium remaining in the bath than if vanadium only were added.

Cerium is also said to be of beneficial influence in aluminium production, and to increase the toughness of pure aluminium by reducing the silicon contents. It is claimed that an addition of 0.2% cerium will reduce a

silicon content of 0.1% to 0.02%. For this purpose cerium should be added to the aluminium in the fused state, preferably in the form of a high cerium containing aluminium alloy, or in the form of cerium fluoride to the electrolytic bath in which aluminium is prepared.

With the exception of flints the only known process in which cerium is used as an alloying element remaining in the finished product is copper alloy containing between 0.1 and 1% cerium, 5 to 10% aluminium, and balance copper. According to an American invention, this alloy has superior characteristics over other metals at present used in bearings, and is particularly suitable for shaftings subjected to severe service and heavy loads. This alloy radiates heat so rapidly that relatively close clearances can be used and are capable of running with the minimum amount of lubrication, or no lubrication at all.

In producing the alloy, chalcopyrite or cerite is used, the iron content in the chalcopyrite is removed by treatment with sulphuric acid. The aluminium and the powder resulting from the treated chalcopyrite are placed in a crucible with copper and heated until the aluminium and copper are fused and have formed an alloy. The cerium also enters into the combination so that an alloy has been produced which is ready for reheating for castings.

Monazite is produced in substantial quantities in Brazil, Ceylon, India and the United States, and is found in small quantities in numerous other countries.

The quantities of thorium oxide required are on the decrease, owing to the gradual disappearance of gas lighting. So far the rare earths metals have been produced as a by-product from the residues, but as increasing employment is found for them it may well be that in the near future the monazite sand will be produced almost solely for the extraction of rare earths metals.

"The Rare Earths," by S. L. Levy.

U.S. Patent No. 1,714,729.

British Unicorn Ltd., 33, Southampton Street, W.C.2.

British Patent No. 302,574.

Austrian Patent No. 246,481.

Annotated Equilibrium Diagrams

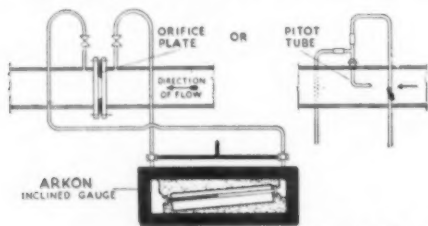
THE Institute of Metals will shortly issue an Annotated Equilibrium Diagram of the Aluminium-Zinc System, prepared by Dr. G. V. Raynor, M.A. The decision to publish this diagram is due to the recognition by the Publications Committee of this Institute of the growing need which exists for up-to-date versions of the equilibrium diagrams of certain binary alloy systems, recent work on which has shown previous diagrams to be in serious error. The Committee has, therefore, provisionally agreed to the issue of a series of Annotated Equilibrium Diagrams, and the aluminium-zinc system has been selected as suitable for the first of these. The Committee would welcome the opinion of members as to the value of such a series, and suggestions regarding the systems which should be included.

It is intended that each of the series shall consist of the diagram in question, reproduced on a generous scale and based on what is regarded as the most reliable work in each phase field; a table giving all important data connected with the diagram; a number of critical notes; and references. The form adopted is that of a folded sheet $11\frac{1}{2} \times 8\frac{3}{4}$ in., which may conveniently be kept in a loose-leaf binder. Members of this Institute can obtain copies of the diagram free of charge by sending a postcard to the secretary, The Institute of Metals, 4, Grosvenor Gardens, London, S.W. 1.

Gas and Air Flow in Gas-Fired Furnaces

An Inclined Gauge Type Indicator for Accurate Draught Readings

FOR the operation of gas-fired furnaces of different types in the engineering, metallurgical and other industries it is often the best policy to install inclined draught gauges having a high degree of accuracy to indicate the gas or air flow in the pipes. An instrument of this kind is the "Arkon" flow indicator inclined gauge type, which reads to one thousandth of an inch w.g. The standard wall mounting type of instrument has a case of substantial cast iron, with brass cover-plate, finished in stoved black-frosted enamel, the whole being screwed to a wall or other support by a 3-hole fixing. At each end of the casing there is a $\frac{1}{8}$ -in. gas union connection together with a cock, these two cocks being coupled by a lever bar and handle, so as to give simultaneous operation. The liquid in the inclined glass tube is red oil, of the same specific gravity as water, the arrangement being fitted in front of an easily readable white enamel scale, with prominent divisions and figures in black, including a screw for zero setting. Also the tube is held at each end in a gland or stuffing box, that is not cemented in, which means it can easily be replaced, whilst the readings are visible at a considerable distance.



Courtesy of Walker, Crosswell and Co., Ltd.

Typical installation of "Arkon" sensitive inclined gauge gas and air-flow indicator for either orifice plate or Pitot tube operation.

In addition there is a similar portable type having a brass case, finished as before in stoved black-frosted enamel. A gauge of this type can be screwed on a vertical surface or alternatively levelled on a horizontal surface by an adjustable three-point support, the cover being held in place by the levelling screw, whilst nipples are provided for rubber tube connections in place of the unions forming part of the wall mounting instrument.

These inclined gauges are used mostly in conjunction with an orifice-plate fitted in the air or gas main and connected by the $\frac{1}{8}$ -in. gas piping. Any type of main can, of course, be equipped, and for 6-in. diameter and over the firm supply a mild steel orifice-plate for mounting directly between the pipe flanges, the two pressure transmitting pipes being taken from each side leading direct to the inclined gauge. For gas or air pipes of less than 6-in. diameter, however, the orifice-plate is supplied in a cast-iron carrier or outer chamber inserted between the flanges, this chamber being complete with $\frac{3}{8}$ -in. gas connections, including drain cocks. The instrument also is in four standard sizes—that is, for operating in conjunction with a 2-in., 3-in. or 4-in. pipe and for any size between 5 in. and 30 in., which, in the latter case, are nearly all fitted, as already indicated, with the orifice-plate between the flanges.

The gauges, however, will work equally well with a Pitot tube device in the main pipe, but for the latter method in place of the orifice-plate it is necessary that the velocity of the gas or air shall be sufficiently high, and for most conditions the orifice-plate is preferable.

The firm also make the well-known "Arkon" gas-flow recorder, which gives a continuous record of the flow of air or gas, as well as indicating instruments of the dial type, but as already mentioned it is sometimes for reasons of economy preferable to use a sensitive inclined gauge.

Some Tensile Shock Properties of Carbon Steels*

THE behaviour of engineering materials under shock loads has been the subject of many investigations. Disagreement abounds amongst the results of these investigations, chiefly because it was, and is, expedient to measure shock or impact in terms of the energy of the shock-producing hammer or tup, and little attempt was made to account for all the energy available. When such an attempt was made the devices employed were only partially successful, owing either to their delicate and elaborate nature or to the severity of the loading imposed upon them.

A machine designed by one of the authors of the present paper has been described in a previous *Journal* of the Institute,¹ together with a preliminary investigation of the behaviour of Lowmoore iron when subjected to both single- and multiple-shock tests. This machine, a simple mechanical device in which elaborate measuring apparatus is eliminated, enables valuable information to be obtained from tests of this nature.

In the multi-shock tests a small amount of residual energy was unaccounted for, and the authors have devised a method whereby the actual energy absorbed by the specimen during such tests can be calculated.

It was suggested that the behaviour of a pure iron under such loading might be different from that of a carbon steel, and the present paper is the result of similar investigations on a family of carbon steels, in which the above machine, together with a simple additional measuring device, has been used.

From a survey of the work carried out, the authors conclude that the following points appear to be of primary importance:—

- (1) The ratio of the single-shock energy to the static energy varies with the carbon content of the steel.
- (2) The elongation of a specimen of carbon steel when ruptured by a tensile shock may be less than the static elongation, while the reduction of area does not vary with the method of testing.
- (3) From the nature of the tensile shock endurance curves there appears to be a limiting range of energy which may be applied an unlimited number of times without causing rupture.
- (4) In a multi-shock test, as the number of shocks to cause rupture increases, the resultant extension and reduction of area increase to a maximum at the same value of energy absorption per shock. Further increase in the number of shocks to cause rupture is accompanied by a rapid decrease in both values of ductility.

* F. V. Warnock and J. B. Brennan. *Iron and Steel Institute*. (Advance copy). July, 1943.

¹ Smith and Warnock. *Journal of The Iron and Steel Institute*, 1927, No. II, p. 323.

Metallurgical Coke

By R. J. Barritt, M.A.

Perhaps one of the most important events in the history of the iron and steel industry was the application of coke as fuel. Its use led to greatly increased sizes of blast furnaces, which in turn necessitated the development of coke possessing suitable properties. These developments are briefly reviewed and reference is made to modern methods of production.

THE term "metallurgical coke," used in its broadest sense, includes all coke used in conjunction with any metallurgical operation—i.e., starting with small-scale operations on the blacksmith's hearth, through ordinary melting operations to foundry cupolas, and thence to the blast furnaces, which are the largest users. It will be seen that a large field is covered and that the term cannot be used with any precision, since a coke suitable for blast furnaces would most probably be regarded as unsuitable for use in the foundry, and so on. Metallurgical coke is frequently known as hard coke, and this gives the first indication of one of the properties required and serves to distinguish it from the softer cokes made in gas retorts and the domestic cokes made in low-temperature carbonisation processes. For all normal purposes hardness—i.e., resistance to both impact and abrasion—the quantity of major impurities, such as water, ash, sulphur, and occasionally phosphorus, the lower size limit or both upper and lower size limits, is sufficient to define metallurgical cokes. Ease of burning—i.e., the ease with which the carbon in the coke combines with the oxygen in the air—which might be expected to be of primary importance, is not usually defined. An easily combustible or "reactive coke" would normally be regarded with some suspicion, since ease of combustion is usually associated with lack of hardness on the one hand and the presence of volatile matter of the original coal, which has not been driven off in the carbonising process, on the other. (A metallurgical coke, therefore, even in suitably small sizes, would not be expected to burn in the ordinary type of domestic fire, but it will burn in special designs of open coke grates arranged to give a reasonably deep fuel bed, or in closed domestic stoves and boilers.)

The properties required above have developed together with the size of the units employing coke. The foundry cupola uses a fairly deep bed of coke and the blast furnace a very deep bed, and it is the capacity of the metallurgical coke to carry the burden in these units and to take the rough handling without undue disintegration, which makes the modern high capacities feasible.

Metallurgical coke is the direct descendant of charcoal, and the development of the use of coke to replace charcoal was a necessary pre-requisite to industrial development, since any large-scale iron industry based on charcoal is clearly impossible. The credit for the first successful use of coke is usually assigned to the Shropshire ironmaster, Abraham Darby, of Coalbrookdale, in the early seventeen-hundreds. Not surprisingly, Darby's technique of coke-making followed that of the charcoal burner, the coke being made by restricted combustion of a pile of coal, and a yield of coke of about one-third

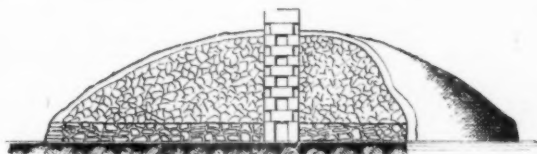


Fig. 1.—Coking in piles. Charge about 50 tons, coking time, 10–12 days.

of the original coal being obtained. Darby used a low-sulphur coal, which would nowadays be considered non-coking, carbonised the coal in lump form and obtained a weak coke, each piece being a portion of the original lumps of coal since these would not coalesce in the coking process. The coke itself would be fairly reactive (easy burring), and this would have been an advantage, since the early furnaces were limited to about 30 ft. maximum height by the capacity of their water-driven blowers, and a reactive coke would be necessary to complete the reduction of the iron ore in the comparatively short travel. The choice of a low-sulphur coal for coke manufacture was of some importance, since the furnaces of Darby's days (and for a hundred years or so afterwards) used a cold blast, and the sulphur content of the iron would tend to be high, making it difficult to work when hot and preventing the manufacturing of wrought iron by hammering. This disadvantage would not be met with when using charcoal, since the sulphur content of charcoal is very low.

Darby may have been fortunate in his locality and choice of coal, but his success was the first step towards large-scale iron manufacture in large units. The charcoal blast furnace made not more than about 10 tons of iron per week, and the early coke furnaces did two to three times this quantity. No further development was possible until the steam-engine driven blower was introduced at the end of the eighteenth century, allowing taller furnaces to be built and a less reactive coke to be used, due to the greater available length of travel of the gases through the burden. With the arrival of the hot blast at the beginning of the nineteenth century wider hearths could be used in the furnaces, and the size of the furnace considerably increased.

Darby's use of a virtually non-coking coal for his coke-making had the rather curious effect of concentrating early coke-making and iron-making in Shropshire, South Staffordshire and Derbyshire, in areas where coke-making is now practically non-existent because of the unsuitable nature of the coal. Hence an area which once held almost the entire English iron industry has now only a very few blast furnaces, although many of the subsidiary industries linked up with the early wrought-

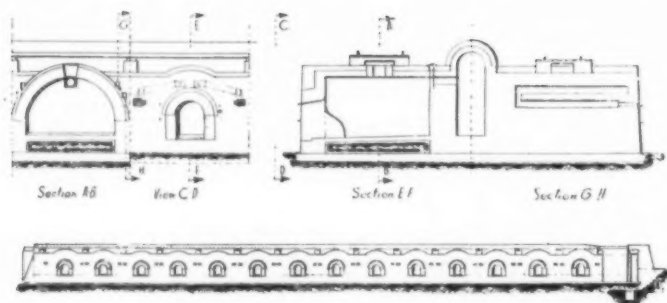


Fig. 2.—Beehive coke oven.

iron manufacture still remain in force in the "Black Country." No doubt the early methods of coking in open smoking piles assisted this district in acquiring its name. Pile-coking (Fig. 1) can still be seen very occasionally in this country and is used in India in the Jharia district to make what is accurately described as "soft coke" in some quantity—but not usually for metallurgical purposes.

Pile-coking was suitable for weakly coking coal in lumps, but unsuitable for the more strongly coking coals of a more swelling nature, and a different technique was developed, particularly in the Durham area, which made a better coke and was able to use the small coals which up to then had been wasted at the collieries. This was the "Beehive" oven (Fig. 2), in which a layer of small coal was coked on a horizontal brick floor by the heat from the arched roof of the coking chamber, under which the gas and volatile matter released from the coal burned. The circular shape and arched roof gave this first coke oven its name, which it retained even when the ovens were built adjacent to each other in batteries. The gas, by-products and part of the coke were still wasted, and the coke yield was about 55% of the coal charged to the oven. The coke (Fig. 3) was hard, columnar in structure, silvery grey in appearance, and unreactive, well suited for use in deep beds, and resistant to breakage in handling and abrasion in use.

The Beehive oven came into its own about 1850, when the North-East iron industry was being founded, and about 14,000 ovens were in existence in Durham some 20 years later. The Beehive ovens produced an excellent coke and had a profound effect on blast-furnace practice generally, enabling tall furnaces with a comparatively high rate of drive to be used. The Beehive oven held almost undisputed sway for about 50 years, and was then gradually ousted by the by-product oven, the forerunner of the modern coke oven. To-day the Beehive oven is something of a rarity, although some beehive coke is still made for special purposes—e.g., crucible steel-melting requiring a high temperature for which the unreactive beehive coke is well suited.

Parallel with the development of beehive coke in Durham and Yorkshire, the use of the hot-blast enabled coal to be used in blast furnaces in the Midlands and in Scotland, and with the higher temperature obtained in the hearth sulphur could be removed in the slag. The coal used had about the same characteristics as that used to manufacture the pile coke which it replaced. The coal-charged furnaces of the Midlands gradually went out in face of competition from the coke-charged type, but in Scotland coal was used until quite recently.

Fig. 3.—Coke from Beehive oven.



The developments in the iron and coke industries given so far have been almost entirely English in origin, but the by-product coke oven has been almost entirely a Continental (and, later, American) development subsequently taken up in due course by England. One reason for this is that the Beehive oven, which made a satisfactory coke with the medium and medium-high volatile Durham coals, did not give very satisfactory results with the Continental coals of lower volatile content, which required more rapid methods of heating. The French and Belgian engineers, who were responsible for the early developments, wanted to heat the charge from both sides, and therefore changed its position from the flat horizontal to the vertical in their newer oven, and placed heating flues on either side. The gas burned in the heating flues came from the coal itself and originally passed through holes in the wall over the top of the coal charge, and burned downwards in the heating flues. The next stage was to add the by-product plant to remove tar, ammonia and benzol from the gas before returning it to the ovens and the original by-product oven had arrived. This happened about 1880, and a number of these "French" ovens were built in this country by Simon Carves about 1882 to 1885, one of which, at Crook, Co. Durham, is still in existence and working, and is probably the oldest battery of by-product ovens in the world.

The by-product oven arrived at a time when iron manufacture in Germany was expanding rapidly and when the output of other countries had become more stabilised. By far the largest proportion of the early ovens therefore was built in Germany, and most of the next development took place there and then spread to other countries. All the fundamentals of the by-product oven were present by 1880, and modifications since that date are refinements—but most important refinements. The addition of regenerators to preheat the air for combustion was the first step towards obtaining better heating efficiency, originally in regenerators common to the whole battery of ovens and then in individual regenerators for each oven. This released about 60% of the gas made for use elsewhere. Improved methods of supplying gas to the heating flues were evolved, enabling more uniform heating to be obtained. Mechanical pushing of the coke from the oven had to be adopted when

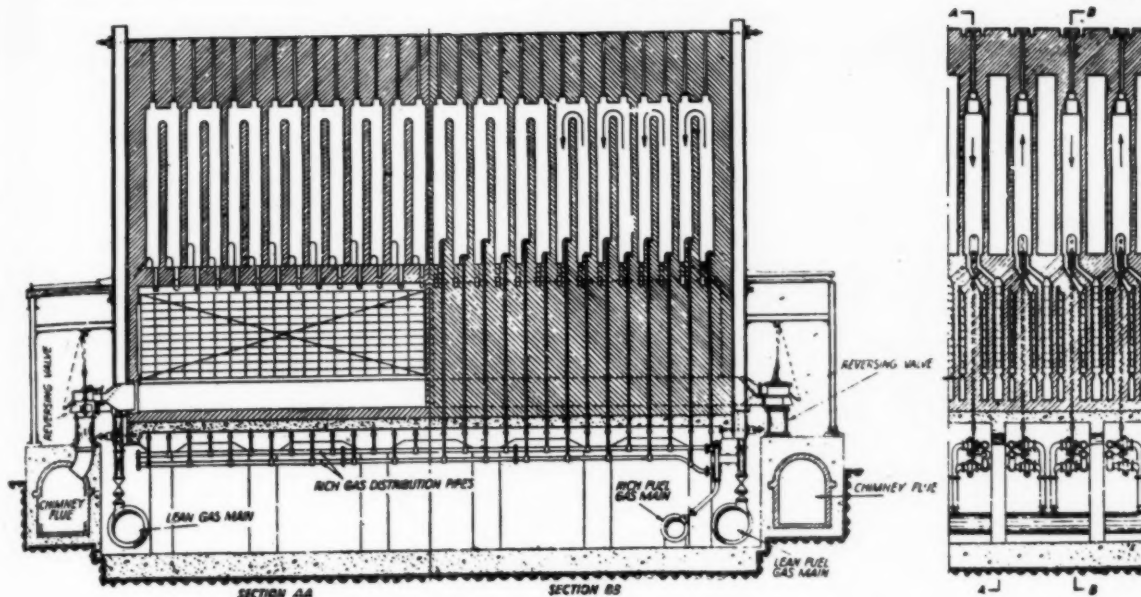


Fig. 4.—Typical modern coke ovens. A Simon-Carves Compound "Underjet" oven.

the coke oven took its present shape, and this was followed by mechanical charging of the coal into the ovens by a charging car operating from a central coal bunker and running along the top of the ovens. In a number of cases the ovens were heated by blast-furnace gas or producer gas, thus releasing the whole of the coke-oven gas made for use elsewhere.

The general excellence of the beehive coke made progress in England slow, since it remained to be proved to the ironmasters that the newer coke was no worse than the beehive coke in terms of consumption at the furnaces on the one hand, and could be produced more economically on the other. Although the first by-product ovens were installed here in 1882, the progress made was slow, and not until about 1905 onwards did the majority of English ironmasters begin to accept the new coke. By 1914 about two-thirds of the coke output of the country came from by-product ovens, and at the present time only about 1% is made in beehive ovens.

The next real progress took place in America, which, up to 1914, had followed a general line of progress similar to that outlined for England. The war vastly increased the demand for steel, and coke demands also went up considerably, with the result that many new coke plants were built. Larger outputs were obtained by using silica brick refractories instead of fireclay or semi-silica, thus enabling higher temperatures to be used, giving a faster coking time and an increased output per oven. At the same time, the size of the oven was increased, with a corresponding effect on the output. The original ovens had a throughput of coal of about 2 tons per day, the European version in the period 1914-1918 had reached about 10 tons a day, and now American practice raised this to about 20 tons. At the same time the mechanical equipment was much improved, particularly in the handling of the coke, which was quenched centrally and then put through a screening station, and delivered to wagons or to the furnace bins by belt conveyers.

In the period 1922-6 the coke oven reached its present form, and though continuous improvements in equipment and details are taking place, no fundamental alterations have taken place since that date. A typical modern oven is shown in Fig. 4, arranged for heating with coke-oven or blast-furnace gas as desired. The oven chambers are about 45 ft. long by 14 ft. high, and a mean width of 18 in., the oven usually being tapered from 17 in. at the pusher side to 19 in. at the discharge side, to assist the pushing of the coke. The heating flues lie on either side of the oven chambers, thus dividing them from each other, and any number of ovens up to about 70 may be located in one block or battery. Under each oven is a regenerator chamber divided into three portions longitudinally and filled with slotted chequer bricks. When the ovens are fired with coke-oven gas all three compartments of the upstream regenerator preheat the combustion air before it enters the heating flues. The coke-oven gas is supplied to the base of the heating flues through channels in the main supporting brickwork pillar. When blast-furnace gas is used to heat the ovens this is preheated in the centre division of the regenerator while the two outer divisions preheat the air, the gas and air then meeting at the base of the heating flues. In either case, combustion takes place in alternate flues and the products of combustion pass down the neighbouring flue and into the downstream regenerator, all three divisions of which abstract heat from the waste gases before they go to the chimney flue. About every half hour the direction of heating is reversed and the upstream and downstream regenerators changed over.

The heating walls and upper parts of the regenerators are built in 95% silica shapes and an operating temperature of 1,500° C. or less is permissible in the heating flues if required. The temperature actually used depends upon the type of coke required and will not normally exceed 1,350° C. for blast-furnace coke and will be less than this for foundry coke.

The first fundamental of good oven heating is to obtain simultaneous completion of coking over the length and

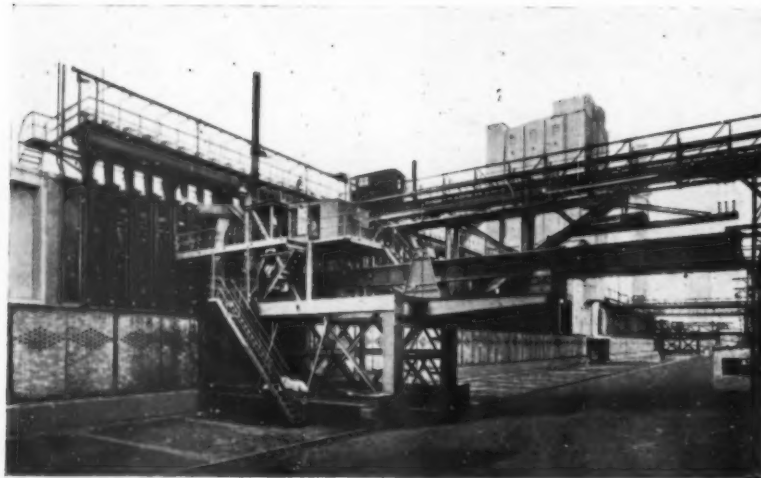


Fig. 5.—Modern coke-oven plant. View on pusher side, showing coke pusher machine.

height of the coal charge, on the one hand because heating a part of the charge already coked wastes gas, and on the other because the quality of the coke itself suffers if it is allowed to "soak" in the oven. Since the oven is tapered from the pusher side to the discharge side this entails a gradually increasing flue temperature over the length of the charge starting from the pusher side, so that coking at the wide end of the oven is completed at the same time as at the narrow end. Somewhat more gas will also be needed at the oven end flues, to compensate for radiation losses, and it will be seen that one of the designer's problems is to arrange his heating system so that the fuel gas supply to individual flues is adjustable. Another point is that the heating in a vertical direction must be uniform, so that the top of the charge is carbonised at the same time as the bottom. This means obtaining a uniform temperature over a 14 ft. high wall when heating either with coke-oven gas (about 500 B.th.u. per cub. ft.) or blast-furnace gas (about 100 B.th.u. per cub. ft.). Blast-furnace gas burns with a long non-luminous flame and offers no real difficulty, but the richer coke-oven gas burns with a shorter flame and tends to overheat the lower part of the flue unless special devices are used. These may comprise varying the height of the burners, admitting air in stages or dilution of the rich gas with flue gases according to the preference of the designer, and in most modern ovens the system is so arranged that a variation of not more than about 30° C. exists over the height of the wall.

So far we have considered the method of carbonising rather than the material being carbonised. The original method of carbonising in piles used a high volatile coal of a non-coking type and around 80% carbon content on a dry ash-free basis. The beehive oven was at its best with the medium and medium-high volatile Durham coals 85–89% carbon content. The by-product oven can carbonise a wider range of coals than the beehive oven, 83–91% carbon content, and the technique of blending enables a fair proportion of coals previously considered unsuitable to be used. It may be noted that the modern by-product oven, as distinct from its

predecessors, uses a finely ground coal (about 85% through $\frac{1}{8}$ in.) to obtain a stronger coke.

Given a suitable type of coke oven, the variables affecting the coke quality are: (1) The nature of the coal charged to the oven; (2) the oven dimensions, more particularly the width; (3) the carbonising temperature. Of these factors, the first and third are to some extent variable after the job has been built, but the oven dimensions must be chosen at the beginning. The oven width used has varied from up to 24 in. in the earlier ovens down to 14 in. in some cases during the middle 1920's, and then has more or less stabilised itself over the range 16 in. to 18 in. mean width for blast-furnace coke plants, and 18 in. or 19 in. width for foundry coke plants. Narrow ovens make smaller coke, and the 14 in. ovens

though given a large boost in this country about 1925–7 made too small a coke for the liking of most blast-furnace managers, and gave a larger proportion of coke below the blast-furnace lower limit (1 in.—1½ in.) than the wider ovens. The narrow ovens were not used for foundry coke-making, since most foundries use a larger and blockier type of coke than is obtainable with a narrow oven and a short coking time.

The temperature in the heating flues affects the speed of coking—e.g., a flue temperature of about 1,350° C. will carbonise an 18 in. wide oven in about 18 hours, and 1,400° C. in rather less than 16 hours, either of these being suitable for blast-furnace coke manufacture. For foundry coke the same oven would have a coking time of 24 to 28 hours and a flue temperature of about 1,150°–1,200° C. A fast coking time yields a higher proportion of small coke and the tendency for blast-furnace purposes in recent years has been to keep to an average rate of 1 in. an hour—i.e., 18 hours' carbonising time for an 18 in. wide oven, etc. Both width and carbonising time have to be chosen to suit the coal—e.g., certain coals which will give practically all small coke in a 14 in. oven with a rapid coking time will give a reasonable coke in an 18 in. oven with a coking time of 22 hours. Set rules, therefore, cannot be laid down, and previous experience, often combined with small or large scale tests, is usually the settling factor for both oven width and coking time. It will be noted that since foundry coke needs a longer coking time than blast-furnace coke, more ovens will be needed for a foundry coke battery than for a furnace coke battery of the same capacity, and the capital charges for foundry coke manufacture are higher than for blast-furnace coke.

The properties of the coal have been left to the last, but are in fact the most important, since if the coal is non-coking or only weakly coking all the other factors are unimportant, as a suitable coke will not be made in any case. A coking coal is a most important national asset, and it is difficult for a nation without proper reserves of coking coal to develop its heavy industries—e.g., Italy is handicapped in this way, and Japan is largely dependent on the supplies from the Kailan mines

in North China: an inducement for the invasion of this area. Indian coking coal reserves are said to be sufficient only for another 50 years at present rates of use, which is a rather serious handicap to a country developing a comparatively large iron and steel industry.

The reason why some coals coke and others do not is not known with absolute certainty, although a fair amount of information exists about the coking process itself. We have already seen that the carbon content on a dry ash-free basis lies between 83% and 91%, and the hardest coke is given in the range 89 to 91% carbon (and hydrogen 4.6 to 5%). The coal as charged to the ovens will normally be crushed, so that the major part is minus $\frac{1}{2}$ in. Even then, about 30% of the charge will be free space, and it is evident that the individual coal particles must expand by at least this amount, otherwise the particles cannot agglomerate to form a coke. As a corollary to this, too much expansion during coking is liable to cause damage to the coke-oven walls, and this characteristic is apt to be possessed by some of the coals in the 89 to 91% class. A number of empirical tests are used, particularly by the firms building coke ovens, to determine the "safe" coals to use—a somewhat doubtful term, since a coal which might be safe at a slow rate of heating could possibly be dangerous at a more rapid rate. As another point in connection with expansion, the coal having softened and expanded, must then in the process of losing its volatile matter and becoming coke, finally contract sufficiently to come away from the oven walls, otherwise it will not be possible to push the coke from the oven.

The changes which occur during the heating of the coal would appear to be a preliminary decomposition stage in the range up to 300° C., in which small quantities of primary oils may be released, followed by a stage when gaseous and liquid hydrocarbons appear with the coal becoming plastic and the individual particles beginning to swell (about 400° C.). During this stage the free space in the coal charge is enclosed by the swelling particles and the pores are formed. As the temperature continues to rise, the plastic coal begins to solidify and the volatile matter is lost, causing shrinkage cracks. In the oven, therefore, where the charge is heated from both sides, a "plastic layer" travels inwards from either wall as the carbonising proceeds, separating the coke from the yet uncarbonised coal. At the end of the carbonising period the plastic layers meet, and there is a final evolution of gas. There is a line of weakness where the two plastic layers have met at the centre of the charge, and the coke charge divides in the centre when the coking is complete. This means that the maximum dimension of the coke-pieces in the direction of the width of the oven is rather less than half the width of the oven.

The swelling of the coal seems in the main to be due to gas pressure caused by the liquids sealing the gas outlets, and the pressure caused may be considerable.

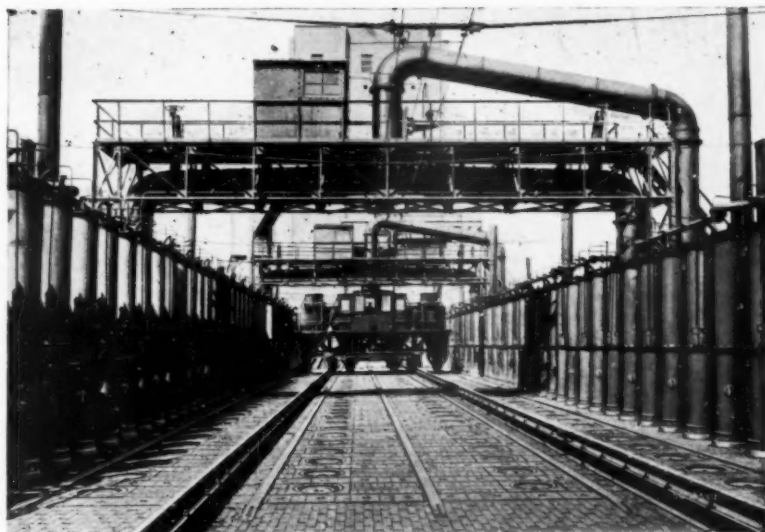


Fig. 6.—Oven tops with coal-charging car.

It is known that non-coking coals have a larger internal surface than the corresponding coking coals, both from direct measurements of the heat of wetting, as in the experiments of Bangham and his colleagues at the B.C.U.R.A., and from other indications, such as the higher inherent moisture content of non-coking coals. It may be, therefore, that the internal surface of the non-coking coals is too large for effective wetting, and that sufficient pressure cannot be developed in the carbonising process to give a coherent coke. On this reasoning the coals which should form the strongest coke would be the high-rank coals, say, 87 to 91% carbon content, having a small internal surface and sufficient volatile matter, indicated by the hydrogen content, to wet this surface. These are the coking coals with the lowest volatile matter, and the shrinkage cracks in the coke will be at a minimum. As the carbon content of the coal decreases the internal surface rises and the limiting hydrogen content for a coking coal also rises. It should then be possible to indicate from the carbon-hydrogen relation whether a coal falls in the coking class. Mott, at Sheffield University, has done a great deal of valuable work on the subject of coal classification and has succeeded in including most of the British coals in one comprehensive classification relating the composition of the coal with a number of empirical tests which serve as an indication of the coking properties of the coal. In our present state of knowledge, however, use is made of the various classifications and tests, but the final decision either on a coal or a blend of different coals, is usually made on a small scale test in a test oven or a full-scale test in an existing plant.

While the method of carbonising plays its part in extending the range of coals treated, most of the desired properties in the coke must be obtained by choice of the right coal or blend of coals. For example, the higher volatile coking coals would not be expected to give as strong a coke as some of the higher rank coals, and might be improved by blending with a proportion of a low volatile coal or even a proportion of non-coking



Fig. 7.—Discharge side of coke ovens, showing hot coke car and coke wharf.

coal or an inert body, such as finely ground coke breeze. Similarly, a good but potentially dangerous swelling coal can be made safe by blending it with a coal of lower rank. Coal blending has been very much to the fore in recent years, but as the results cannot always be prophesied even when the properties of the individual constituents are known, it is usual to carry out experiments to improve the results of the shatter test of the coke and the Cochrane or abrasion test results.

These tests have been devised to give an indication of the behaviour of the coke in relation to the two main methods of disintegration—viz., impact shock by being dropped on its way to the point of use and wearing away by rubbing. Abrasion takes place mostly in the shaft of the furnace or cupola in distinction to breakage by impact, which takes place mostly before the furnace is reached. The shatter test comprises giving 50 lb. of coke over 2 in. in size four 6-ft. drops on to an iron plate, and then screening the resultant coke to determine the percentage remaining on a 2 in. or $1\frac{1}{2}$ in. screen. These percentages are known as the 2 in. and $1\frac{1}{2}$ in. shatter indices respectively, the latter being the one most generally used. The Cochrane test consists in rotating 28 lb. of coke 2 in. to 3 in. in size, in a 30 in. diameter drum for 1,000 revolutions at 18 r.p.m., and then determining the percentage remaining on a $\frac{1}{8}$ in. mesh screen. Both these tests arose as the result of the practical experience of coke users, which showed that an excessive amount of smalls or dust in the coke caused a decrease in the capacity of their furnaces and an increase in the amount of coke used per ton of product. As might be expected, the lower volatile South Wales and Durham coals give the higher shatter indices with the more volatile Yorkshire coals somewhat below. The Cochrane abrasion index is influenced by the amount of inert matter present—e.g., a high-ash coal usually gives an abradable coke. Similarly, addition of finely ground coke breeze to a medium-high volatile coal with the object of improving its shatter index may achieve its

object, but at the same time make the coke more abradable, and the final result may be more undesirable than the original.

Properties other than physical, such as ash and sulphur contents, also have their importance. Thus, for cupola work a sulphur content in the coke less than 0.8% is desirable, also as low an ash content as can reasonably be obtained, so that the slag quantity can be kept down. For these two reasons foundry coke ovens usually take a special fraction from the coal-washing plant, and this combined with the slower coking time adopted in the manufacture to give the rather larger and denser product than for blast furnaces, explains the extra cost involved.

The amount of ash and sulphur permissible in blast-furnace coke varies in accordance with the type of ore. Thus, with lean ores the volume of slag is relatively large, and a low ash content is desirable in the coke (e.g., less than 10%) to keep the volume down. With the rich ores a high ash content is no disadvantage in most cases—e.g., in India, using a rich ore, the ash content of the coke is over 20%, and yet the furnaces have production figures equal to the largest in the world. High-ash cokes are also used in Australia and South Africa, as both are fortunate enough to have rich ores. With rich ores, however, the amount of sulphur carried away by the slag is correspondingly limited, and it is not unusual to fix a limit of about $1\frac{1}{2}$ % sulphur in the coke. Lean ores, on the other hand, can manage up to about $2\frac{1}{2}$ % sulphur without undue worry.

Finally, it will have been noted that the best grades of coking coal are a wasting asset, and the time may come when their use for purposes other than metallurgical coke manufacture will have to be restricted. The alternative is a different type of process for iron-making. Up to date, this has not been developed, but no doubt the need will be adequately met when the occasion arises.

Gas Cyanisation*

A gas for simultaneous nitration and carbonisation (nitrocementation) must be rich in carbon and must contain at least 50% hydrocarbons. It is necessary that the cementation gas and the NH_3 be brought to the furnace in separate lines so that mixing takes place only after the gases are in the furnaces. Cyanide compounds are formed in the nitrocementation process. For this reason care must be taken that no gas leaks from the furnace into the surrounding working space. With a temperature increase from 600° to 830°C ., and a retention period of 90 mins., or with a temperature of 830°C . and a period of cementation of 1–4 hours, the curve for the depth of saturation of the steel with carbon and nitrogen approaches a straight line. The nitrocementation process gives equally good results with straight carbon steel and with chromium-molybdenum and chromium-manganese-molybdenum steels.

* N. F. Vyaznikov and A. A. Yurgenson, *Metallurgy*, 15, 24–32.

Progress in Micro-Testing

By D. W. Rudorff, M.Inst.F.

The method of micro-testing is not yet generally appreciated, but it may prove to be an invaluable factor in raising present-day comparative physical test methods to a more scientific level. Present-day testing methods are criticised by Chevenard, and this review of his recent work on the subject will be of interest.

IN principle the study of the thermoelastic properties of a metal by means of a static method appears to be a most simple matter: A load imposed upon a test-piece causes a certain deflection, and the variation of this deflection with the temperature will therefore be a measure of the corresponding variation in the modulus of elasticity. For example, such changes in deflection with the temperature can be measured with a device based on the differential dilatometer principle. The diagram thus derived can then be used for the determination of the thermoelastic coefficient over the temperature range considered.

Unfortunately several factors intervene which make this simple method inapplicable. Actually, the variation with the temperature in the deformation caused by the application of a constant load is the sum total of (1) the reversible thermoelastic effect which is to be studied, (2) a viscous deformation, and (3) a spontaneous deformation of the test-piece caused by the relaxation of internal stresses. Thus, for instance, it has been found that the heat of a wire as it leaves the die of the draw bench is sufficient to cause a noticeable, although minute, torsion.

New Method for Investigating Thermoelasticity

In order to arrive at the accurate determination of the thermoelastic effect alone, the errors introduced by viscous and spontaneous deformation must therefore be excluded. This can be achieved by subjecting the test-piece to an oscillating motion produced by a variation of the load between two equal and directionally opposed forces, instead of applying a constant loading. With this method the difference in the amount of deflection produced at different temperatures will then be a sole measure of the corresponding variations in thermoelasticity with the temperature. The apparatus devised by P. Chevenard* to measure the thermoelastic coefficient by means of such an oscillating torsion test, is shown in Fig. 1. In this design no effort is made to effect correction for the influence of temperature upon the dimensions of the test-piece. From known data on the dilatation of materials it would, however, be easy enough to make a correction. But actually no such correction is required in practice.

Referring to Fig. 1, the test-piece *F* is made of a piece of 0.1 mm. diameter wire, 20 mm. in length. It is concentrically arranged in a resistance type furnace (not shown). At its lower end the test-piece is fastened to the moving system *E* carrying the mirror *M* and the small armature *n-s* arranged between the poles *P_n* and *P_s* of the magnet *A*. The latter, revolving on a shaft concentric with the axis of test-piece and moving

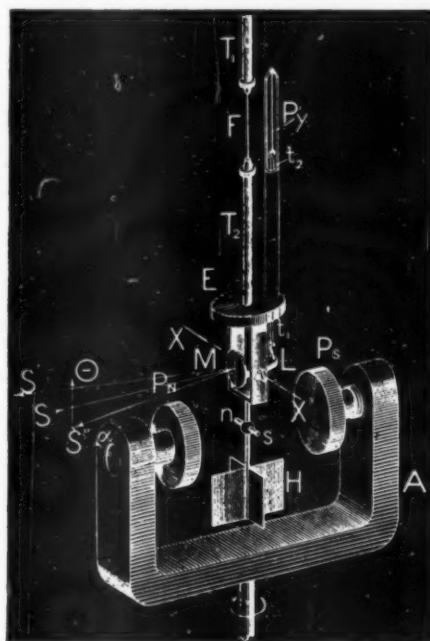


Fig. 1.—
Thermoelasticity
indicator
after
Chevenard.

system, is driven by a clockwork of adjustable speed, which imparts a practically constant sinusoidal turning movement to the system. Temperature compensation is effected by a magnetic shunt of special steel (not shown), while the amplitude of the oscillation is adjusted by varying the gap between the adjustable pole pieces *P_n* and *P_s*. Below the armature of the moving system a damper is arranged which actually is immersed in oil.

In addition to indicating the torsional deflection of the moving system in the horizontal, the mirror *M* is also made to indicate the temperature of the test specimen by a corresponding deflection in the vertical. This is achieved by making the mirror rotatable around the axis *X-X*, the degree of rotation being determined by the expansion or contraction of the dilatometric pyrometer *Py*, to which it is linked by means of the lever *L*. Owing to its proximity to the test-piece, the action of this pyrometer is a highly accurate measure of the temperature of the piece.

The type of diagram actually registered with the apparatus is exemplified by Fig. 2, which is a photographic record of the amplitude of the oscillation versus the temperature of the specimen. From such a diagram the thermoelastic coefficient is very simply derived, as

* *Rev. de Met.*, vol. 39, p. 66.

follows: It is by definition $\gamma = \frac{1}{\mu_0} \cdot \frac{d\mu}{d\theta}$, where γ is the thermoelastic coefficient, μ is the Coulomb modulus, and θ is the temperature. Taking a and x as the axis of the chart given in Fig. 2, the ordinate a represents the arc $A + \epsilon$, while the abscissa x is a known function of the temperature.

If $\frac{da}{dx}$ is the differential quotient at a point x of the abscissa corresponding to a temperature θ , and μ is the Coulomb modulus at this temperature, it can be written

$$\gamma = \frac{1}{\mu_0} \cdot \frac{d\mu}{d\theta} = - \frac{a_0}{a^2} \cdot \frac{da}{d\theta} = - \frac{a_0}{a^2} \cdot \frac{da}{dx} \cdot \frac{dx}{d\theta}$$

The arcs a_0 and a can be obtained from the recorded diagram, and $\frac{da}{dx}$ can be determined graphically, while $\frac{dx}{d\theta}$ is known from the calibration of the pyrometer of the moving system.

By applying this method of interpretation to the graph given in Fig. 2 the thermoelastic curve $\frac{\mu\theta}{\mu_0}$ of

Fig. 3 is obtained. Actually, these diagrams refer to an invar wire which was annealed at 475° C. after drawing. Fig. 3 shows all the known anomalies in the thermoelastic behaviour of the ferro-nickels of the Invar group. The modulus is seen to increase as the temperature approaches the transformation point and the alloy moves towards the paramagnetic state. After this point is reached, that is, from the Curie point (situated at 250° C.) onward, the curve begins to dip. Towards 375° C. an increase in steepness marks the rapid growing of the elastic hysteresis. This point of inversion of the curve $\frac{\mu\theta}{\mu_0}$, exactly coincides with the analogous

characteristic of the $\frac{T_0}{T\theta}$ curve obtained with the dynamic method carried out by means of the Coulomb pendulum.

In another example, related by P. Chevenard, the thermo-elasticity indicator is applied to the study of the thermoelastic anomaly of nickel. Heretofore, the inter-relationship of the Curie point and thermoelasticity has been very difficult of determination by means of the Coulomb pendulum because of the rapid damping at temperatures above 250° C. With the use of the new device this difficulty is, however, completely overcome. A similar condition is experienced with a nickel-chromium alloy containing 2.35% chromium. The thermoelastic characteristic obtained in this case is charted in Fig. 4. Here it is seen that the point of greatest curvature exactly coincides with the Curie point of the alloy, the arrows indicating the Curie point as determined by the thermomagneto-metric method.

That the instrument is equally useful in exploring the thermoelastic anomaly of iron-cementite aggregates is evidenced by Fig. 5. Like every ferro-magnetic substance, cementite possesses a dilatometric anomaly correlative with the magnetic transformation, minimum dilatibility coinciding with the thermomagnetic Curie point. This pronounced negative anomaly is encountered in all iron-cementite aggregates, even in carbon steels with less than 0.1% carbon. Previous methods had not permitted a close study of this phenomenon in the

vicinity of the Curie point of the cementite.* The superiority of the new instrument in this respect is strikingly evidenced by the graph shown in Fig. 5, which was obtained with a 1.18% carbon steel after a 1-hour anneal at 750° C. Here the (corrected) γ value attains a pronounced maximum at the Curie point, this singularity being in exact symmetry with the minimum value of dilatibility. Therefore the carbon steel is affected by a positive thermoelastic anomaly.

Fig. 2.—Thermoelastic diagram recorded for invar wire annealed at 475° C. after drawing.

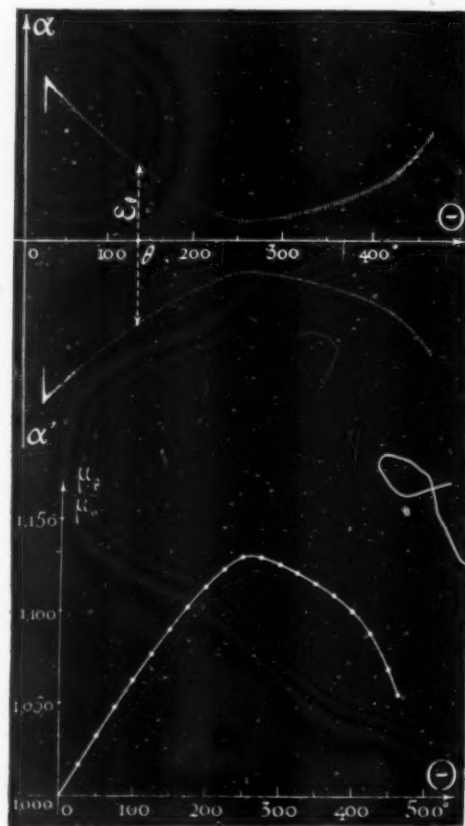


Fig. 3.—Thermoelastic curve $\frac{\mu\theta}{\mu_0}$ derived from Fig. 2.

Micro-testing Technique

It would appear that the method of micro-testing has not yet received the general appreciation which it decidedly deserves. Here again P. Chevenard's work deserves the closest attention, since it may well prove to be an invaluable factor in raising present-day comparative physical test methods to a more scientific level. The unsatisfactory situation as it prevails to-day is especially well described by Chevenard's criticism of present-day testing methods.† Indeed, it would not be easy to answer his contention that in tensile testing, test specimens are too short to allow the existence of pure and uniformly distributed tensile stress. Still less satisfactory are conditions in impact testing, where no

† *Rev. de Met.*, vol. 39, 1942, p. 33.

law of similitude has yet been found to bring different test methods on to a common basis of comparison.

In order to avoid making the preparation of test-pieces unreasonably difficult, a standard test-piece diameter of 1.5 mm. has been adopted by Chevenard in micro-testing. This diameter is still large enough to keep the influence of machining inaccuracies within fairly small margins, a diameter variation of 0.005 mm.

Fig. 4.—Thermoelastic curves and thermoelastic coefficient of chromium-nickel steel. (The arrows indicate the Curie point).

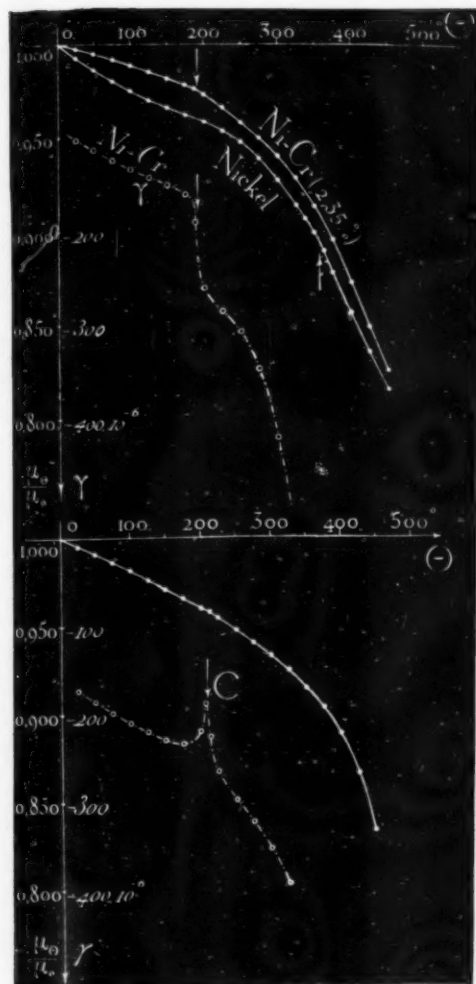


Fig. 5.—Thermoelastic curve of 1.18% carbon steel annealed for one hour at 750°C. (C indicates the curie point.)

producing a deviation by 0.66% from the specified momentum of inertia. For tensile tests a gauge length five times the diameter, that is 7.5 mm., was chosen, while for torsional tests a gauge length of 10 mm. is used. In the thermal treatment of pieces of such a small size special precautions must be adopted—vacuum treatment being by far the best method. Practically equally good results can, however, be obtained with a protective atmosphere of purified argon in the furnace.

The first micro-test device constructed according to Chevenard's plans at the Imphy laboratories of the S.A. de Commentry-Fourchambault et Decazeville, was the tensile and shear test machine, the essential features of which are shown in Fig. 6a and 6b. Here R represents a vertical bar of elinvar arranged as a vertical cantilever, which serves as the straining head, the required test load being produced by the deflection of its upper free end. The opposed lever L is pivoted at its lower end (not shown), and its distance relative to R—and with it the stress imposed upon the piece—is controlled by a

Fig. 6a.—Chevenard's micro-tensile test apparatus.

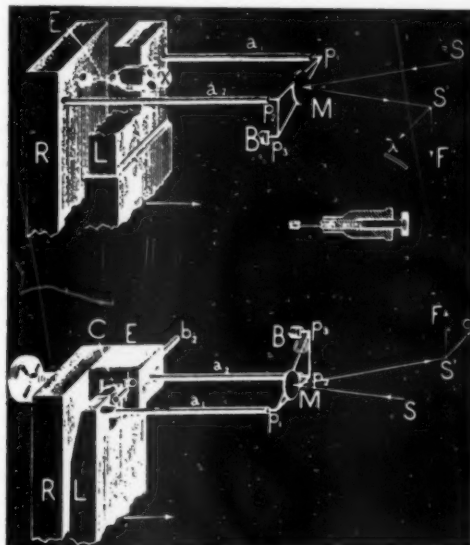


Fig. 6b.—Chevenard's micro-shear test apparatus.

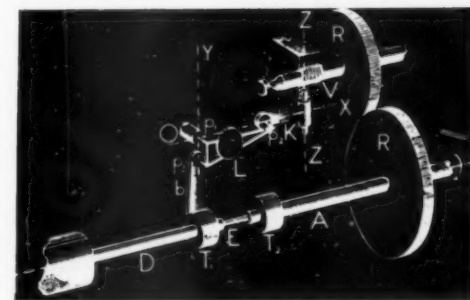


Fig. 7.—Chevenard's micro-machine for static torsion test.

motor-driven worm-gear mechanism. In the tension test, Fig. 6a, the test specimen E is seen to be held by grips provided in R and L. The arrangement used for shear tests, shown in Fig. 6b, consists in fastening the test-piece in a special holder attached to cantilever R; while lever L is equipped with a knife edge. Registration of the movements during the test is effected by means of the mirror-tripod system shown. Thus, in the tension test set-up, Fig. 6a, in which point p_2 of the mirror tripod is fixed, the relative displacement of feelers a_1 and a_2 , caused by the elongation of the sample piece, is expressed by the deflection of the reflected light ray in the direction

of the abscissa λ ; while the elastic deflection of R, which constitutes a measure of the test load, is indicated by the deflection of the reflected beam of light in the direction of the ordinate F of the photographically recorded test diagram. The diagrams obtained with the shear device are analogous. Tests of the latter type have proven of

which is driven by a 25-cycle synchronous motor. Here again, test-pieces of 1.5 mm. diameter and 10 mm. length are used.

A typical diagram obtained in the testing of a chromium-nickel steel with 0.08% carbon 18.7% nickel, and 23.6% chromium is shown in Fig. 10. During the first

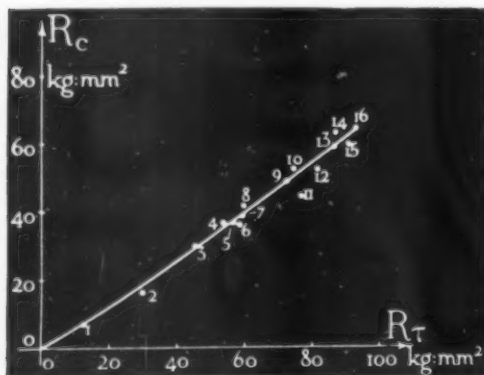


Fig. 8.—Relationship between true shear R_C and torsional shear R_T .

particular value in the investigation of weld seams in which the use of small test-pieces is, of course, essential.

The principal features of the latest machine developed by Chevenard for static torsion testing are shown in Fig. 7. Here again a test-piece (E) of 1.5 mm. diameter and 10 mm. length is used, its ends of 3.5 mm. diameter being held in the grips T_1 and T_2 . Shaft D is stationary, while torsion is effected by rotation of shaft S driven over a reduction gear by a variable speed motor (not shown). Here also a tripod mirror L_4 with the stationary point at p_1 , is used as indicating organ. Since the angular movement of shaft A in the torsion test is considerable, exceeding as it does 10 complete revolutions in practice, point p_2 of the mirror tripod is shifted by a "demultiplicating" device operated from the gears R, R_1 , thus reducing to the required scale the deflection of the mirror caused by the torsional movement. The angular deflection of shaft D, signifying the prevailing torsional loading of the test-piece, is recorded by the mirror deflection caused by movement of tripod point p_2 .

Of particular interest is a comparison of shear strength values obtained from this machine with corresponding values obtained in pure shear tests with the set-up illustrated in Fig. 6b. By charting the respective results obtained with some 15 different steel specimens, and also with one specimen of ordinary aluminium No. 1, Chevenard obtains the relationship shown in Fig. 8, where the true shear stress is seen to closely approximate to seven-tenths of the torsional shear stress. This is by no means surprising, if it is considered that in the torsional test the most highly stressed fibres are supported and kept from failure by less-stressed adjoining fibres.

For the torsional fatigue testing of micro-samples, the working principle shown in Fig. 9 has been adopted. It is quite similar to that employed in the static machine, but because of the smaller working cycles traversed in fatigue testing, the use of the demultiplicating device could be dispensed with. The tripod mirror system in its simplest form can therefore be used, as seen in Fig. 9. In this machine the oscillating movement is simply effected by a crank mechanism with adjustable stroke,

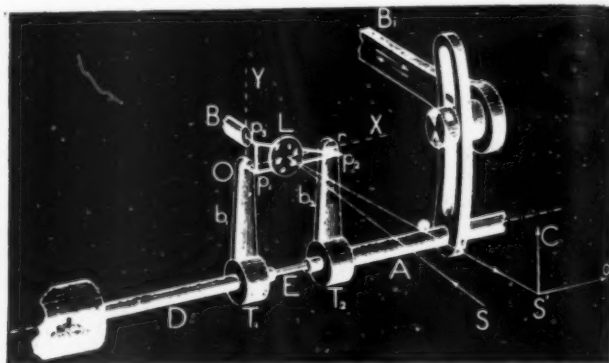


Fig. 9.—Micro-machine for torsional fatigue testing developed by Chevenard.

three cycles at the top the trajectories are merely indicated by straight lines, but from the fourth cycle onward a loop is formed. The hysteresis is thus seen to increase with the amplitude in cycles 5, 6 and 7. It subsequently decreases (cycles 8 and 9), and again becomes invisible in cycle 10. Evaluation of this diagram

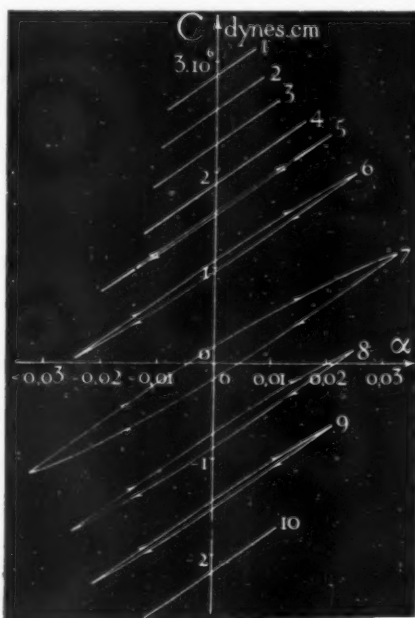


Fig. 10.—"Couple torsion" cycles registered with an annealed micro-sample of 0.08 C, 18.7 Ni, 23.6 Cr steel.

is exemplified in Fig. 11, which refers to the cycle No. 7, recorded in Fig. 10. The $\frac{dy}{dx}$ curve derived from the cycle is shown on top of the latter; from this curve the values of the true modulus μ_r can be calculated for every point of the cycle. The actual values μ_r obtained for the various cycles are charted in Fig. 12. By planimetry

the cycle areas the amount of energy W dissipated by internal friction into heat can be computed. Because of the considerable amount of time involved in the evaluation of cycle diagrams this method of analysis will rather have to be limited to laboratory research. For practical

purposes the usual procedure of ascertaining the limiting safe range of stress is definitely preferable.

In view of the considerable extent to which creep tests are employed in determining the high temperature strength of heat-resistant materials the creep-test device developed by Chevenard for the testing of micro-samples must also be mentioned. The salient features of this device are outlined in Fig. 13. Here it is seen that the tensile loading is applied to the test-piece by means of the two tension rods T_1 and T_2 , made of a high heat-resisting alloy with 0.3% C., 1.15% Mn, 0.2% Si, 60% nickel, 12% chromium, and 2% tungsten. The feelers t_1 and t_2 actuating the optical system are applied to the measuring points with a slight pressure produced by the weighted levers B_1 and B_2 . The mirror itself is horizontally pivoted in points p and p_1 , the deflecting moment being supplied at point p_2 .

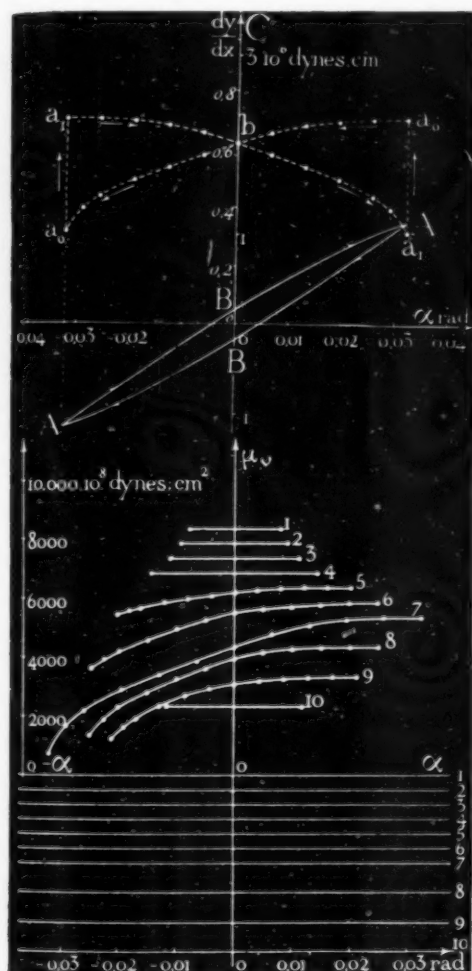


Fig. 11.—Interpretation of cycle No. 7 registered in Fig. 10.

Fig. 13.—Chevenard's micro-machine for high-temperature creep tests.

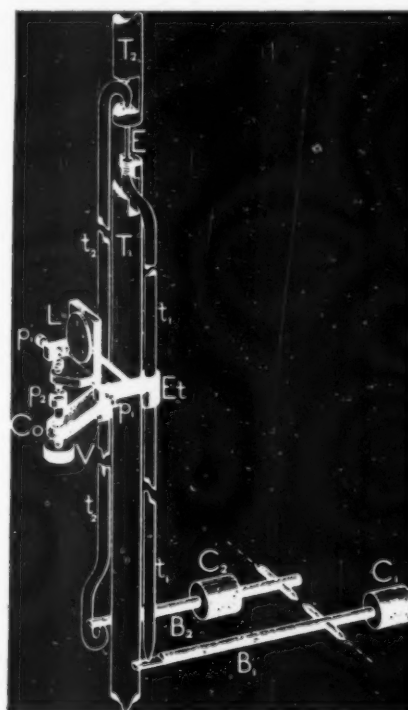


Fig. 12.—Variation of the (true) Coulomb modulus with the torsional angle α over the ten cycles registered in Fig. 10.

A Fire Code for Magnesium Powder Production

WITH the large increase in production of magnesium powder and dust for war purposes there goes a corresponding rise in fire and explosion hazards. Magnesium powder or dust will readily ignite and whilst in suspension in air will explode violently. The high pressure recorded and the very rapid rate of pressure rise observed in laboratory tests, emphasise the importance of adopting all possible protective measures wherever magnesium powder is produced, processed or handled.

At dust concentrations of 500 milligrams per litre (500 oz. per 1,000 cub. ft.) maximum pressures in excess of 60 lb. per sq. in. (4½ tons per sq. ft.) were recorded in laboratory tests. The maximum rate of pressure rise determined was 800 lb. per sq. in. per sec. In

another test, with different apparatus, a rate of nearly 2,600 lb. per sq. in. per sec. was recorded. This means that the maximum pressure of 60 lb. per sq. in. at the concentration cited is developed in from one-tenth to one-fortieth of a second.

The unstable characteristics of magnesium powder and the jeopardy in which life and valuable materials are placed when it is handled incorrectly has been quickly grasped by the Fire Offices' Committee in this country. Resulting from their investigations into the fire and explosion hazards of this manufacturing process is a list of precautions to be observed under the title of "Code of Precautions recommended for Magnesium Powder and Magnesium Alloy Powder Factories." This pamphlet will be sent free on application to the Fire Offices' Committee, 65/66, Watling Street, London, E.C. 4.

A Thermodynamical Theory of Restoration Phenomena in the Ageing of Copper-Aluminium Alloys*

An attempt is made to develop a theory regarding the exact nature of the changes which accompany ageing phenomena in alloys at normal temperature.

IN spite of the numerous investigations which have been undertaken, the exact nature of the changes which accompany ageing phenomena in alloys at normal temperatures (natural ageing) remains a subject of discussion. Recent experimental facts, however, indicate that the original controversy as to the heterogeneous character of the ageing process should be considered from a somewhat different angle. In fact, microscopic analysis (by Fink and Smith, Gayler and Wassermann), and also X-ray investigations (by Preston and Guinier), point indisputably to the conclusion that structural changes do take place in alloys during ageing. Thus, changes in physical properties (hardness, electric resistance, magnetic properties, etc.) are apparently due not only to variations in the state of the atoms, but also to quite perceptible changes in the crystal structure. Accordingly, discussion should be directed to the nature of such structural changes and their relation to variations in the physical properties.

According to Preston¹ and to Guinier,² in the initial stage of the ageing of copper-aluminium alloys, the copper atoms segregate in the form of thin plates, about 2-3 atomic distances thick and 10-15 in. area. These atoms are arranged in a lattice which seems to differ only slightly from that of the solid solution. According to Preston the lattice formed during annealing at 200° C. possesses a tetragonal deformed structure of the CuF_2 type ($c/a = 1.015$). This lattice is oriented with its [001] face parallel to the cube face of the solid-solution lattice, and the [100] direction parallel to the cube diagonal. Thus, the lattice of aluminium atoms in the fluoride structure coincides with the cube face of the parent aluminium lattice. Bradley³ arrived at a similar conclusion in his investigations on precipitation in ternary copper-nickel-aluminium and iron-copper-nickel alloys. Fink and Smith⁴ state that even after ageing copper-aluminium alloys at normal temperatures they detected microscopically straitions, which indicated the precipitation of a phase. Wassermann's⁵ statement that similar straitions can be observed in the alloy immediately after quenching can scarcely be regarded as conflicting with the view that structural changes occur in the alloy even at room temperature; on the contrary, it may be contended that these characteristic straitions cannot appear after quenching without accompanying precipitation phenomena. This view is confirmed by the similarity of the microstructures of quenched and of aged aluminium alloys, which is demonstrated with exceptional clarity by the excellent photographs of Wassermann.

The facts clearly bring us back to the original view of Merica, Waltenberg and Scott⁶ on the ageing of Duralumin alloys as a heterogeneous process, with the difference, however, that the state of the dispersed precipitated phase is somewhat peculiar. This phase is distinguished not only by its structure, which seems to resemble with remarkable closeness that of the solid solution, but also by its form, which is that of very thin plates whose thickness is of the order of a few atomic distances. However, inasmuch as these new formations possess a boundary separating them from the solid solution, it is permissible to call them a phase. On this basis the theory of heterogeneous equilibrium between the phase and the solid solution may be applied.

In the present paper by Professor S. T. Konobeevski, an attempt is made to develop such a theory in a form that should throw light on the behaviour of such systems existing in a state of metastable equilibrium. Thus, the ageing of copper-aluminium alloys is considered as a precipitation process in which crystals of the CuAl_2 phase form in thin plates (discs) in the supersaturated solid solution. As a result of its high degree of dispersion the precipitated phase can exist in metastable equilibrium in slightly supersaturated solutions. Increase of temperature leads to resolution of the precipitated phase; this explains the phenomenon of "restoration" observed during the heating of naturally aged Duralumin. From the temperature of complete restoration the relative amount of the precipitated phase may be determined as a function of temperature and total alloy concentration.

It is found that the *dimensions* of the equilibrium nuclei are independent of the total concentration. However, the *number* of precipitated crystals depends on the initial concentration of the solid solution, since this concentration determines the critical magnitude of the nucleus, and hence the energy of formation of the phase. It is assumed that the number of grains formed depends exponentially on their energy. This enables an estimate to be made of the magnitude of the precipitated crystals and of the value of the surface energy of the boundary between the precipitated phase and the solid solution.

The dimensions of the equilibrium crystal formed during the ageing of copper-aluminium alloys at normal temperatures have been determined (the diameter $2r \approx 10^{-7}$ cm., the thickness $d \approx 3 \times 10^{-8}$ cm.), the values obtained being near to those found experimentally by Preston and by Guinier and his co-workers.

For 5% copper-aluminium alloys the number of crystals in the precipitated phase is 1.3×10^{20} , and for 2% alloys 0.5×10^{20} per gm. The specific surface energy is found to be $\sigma = 0.75 \times 10^{-6}$ cal./cm.²

* *Jour. Inst. Metals*, 1943, Sept., pp. 397-413.

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2 A. Guinier, *Compt. rend.*, 1937, **204**, 1,115; 1938, **206**, 1972.

3 J. Culvet, P. Jaquet and A. Guinier, *J. Inst. Metals*, 1939, **65**, 121.

4 A. J. Bradley, *Proc. Phys. Soc.*, 1940, **52**, 80.

5 W. L. Fink and D. W. Smith, *Trans. Amer. Inst. Min. Met. Eng.*, 1936, **122**, 284.

6 G. Wassermann, *Z. Metallkunde*, 1938, **30**, 62; *Proc. Phys. Soc.*, 1940, **52**, 96.

6 P. D. Merica, R. G. Waltenberg and H. Scott, *Sci. Papers Bur. Stand.*, No. **347**, 1919.

Research in the Ferrous and Non-Ferrous Industries

The Organisation of Research Associations

British industry will be faced with immense tasks after the war, and if it is to maintain its position in the world's markets greatly increased attention must be given to research. Competition for world trade will be keener than ever, and the success of British exports will depend upon the merits of the products offered. There is a tendency to give more thought to political and financial aspects of the export trade, and too little to a consideration of the exports themselves. A vital necessity is more research, which can only be effected by a change of attitude of British industry as a whole towards research. Here reference is confined to co-operative research organisations that are capable of substantial development, but the demands and support must initially come from the industries concerned.

SO few have had a scientific education that there is a tendency to regard research as something remote, difficult and highbrow, in which the many have little direct concern. Actually, research is a habit of mind which makes us attack every problem, big or little, in an orderly systematic way, using, if possible, the advantages that modern science can give us. In industry Britain possesses the advantages of skill and traditions of her craftsmen, but in the past problems encountered have only been possible of solution by careful scientific investigation, and in order to re-fashion existing industries and create new ones, to maintain full employment for these craftsmen, industry will depend more and more on the science that directs its efforts. The post-war period will be difficult, and will raise many industrial problems of far-reaching importance to the future of this country; there is no reason why these problems should not be solved by scientific research, provided it is adequately supported.

It has been said that many competitor countries utilise the services of science in the development of industry to a far greater extent than we have in Great Britain, and though it is difficult to make comparisons, there can be no doubt that, despite some outstanding exceptions, there has not been a general appreciation of the value of science in industry, with the result that the amounts spent on research have been correspondingly less. Yet, as Mr. W. C. Devereux pointed out a few months ago, if only industry and the Government could be persuaded to increase their contributions to established associations ten times the amount at present subscribed we might begin to get profitable results. In the non-ferrous industry this would amount to about half a million pounds per annum, or about half of 1% of the value of the output of the industry in 1935. The increased investment would promote greater interest from member-firms, and they would certainly begin to show demands for results that are essential to give the necessary drive.

In the early days the evolution and development of ferrous and non-ferrous metallurgy was based on practical experience, and occasionally masterly insight, rather than upon extensive learning. Gradually, however, scientific and technical societies and institutions began to be formed, beginning with the Royal Society in 1660. Generally, these organisations consisted of

varying numbers of men who were enthusiastically interested in advancing a particular branch of knowledge, generally associated with their daily task. The spirit of mutual help and emulation, together with a strong desire to advance the subjects they were interested in, constituted the sole motive force. By focusing existing knowledge and honouring outstanding contributions of knowledge in their particular field of interest, these societies and institutions made a major contribution to the means of giving the British people that increasingly high standard of existence. Since those earlier days these societies and institutions have increased and continue to function with success and advantage to the general community. Indeed it is hoped that whatever changes the future may bring, the nature and function of these organisations, so fundamental to national progress, may be preserved.

Great industrial developments usually depend on fundamental discoveries; thus the electrical industry grew from Faraday's researches on electromagnetic induction, and the radio industry sprang from the work of Hertz on electromagnetic waves carried out in a university. It is in the university laboratories that fundamental research is mainly done. At any time a new industry may be born there which will meet some human need. It is in the atmosphere of the university laboratory, too, that the men on whom we have to rely as leaders of industrial research are trained. Industry, therefore, has a direct interest in seeing that research at universities and in technical colleges is adequately endowed.

Another group of establishments at which much research work is carried out are the research stations of the Department of Scientific and Industrial Research. These now consist of the National Physical Laboratory, the Fuel Research Station, the Building Research Station, the Forest Products Research Laboratory, the Road Research Station, the Water Pollution Research Laboratory, and several stations under the Food Investigation Board. The National Physical Laboratory, which was originally established for the maintenance of national standards, is now organised into a group of departments which carry out research, mainly of a fundamental character, directed to industrial needs. Some work is also carried out for research associations.

Another important group concerned with industrial

research are the research associations which were started at the end of the last war with the help of a million-pound fund voted by the Government to the Department of Scientific and Industrial Research. There are now 24 of these associations, and they are self-governing bodies, supported voluntarily by firms engaged in the industry for which they cater, or in a few cases by a levy on the raw materials of the industry in question. The Department of Scientific and Industrial Research supports the associations financially by means of annual grants to an amount depending upon the subscriptions from industry. Although most of these associations have steadily expanded in size and usefulness, they are not yet on a scale commensurate with the industries they serve. One of them is now planning to spend £200,000 per annum, but only four of them have an expenditure exceeding £50,000 per annum, which represents 0.06% of the turnover of the industries concerned. This is obviously insufficient to meet the needs of post-war problems. While there is much to be said for maintaining the voluntary character of these associations, it is apparent that many firms make no contribution whatever, and the time cannot be far distant when a levy on turnover, or on profit, will be regarded as the only fair method of contributing to the essential research services of the industries concerned.

Apart from private industrial research laboratories these research associations have the most direct contact

with industry, and in the following articles the activities of two of the organisations which are included in this group are outlined. The first concerns the British Non-Ferrous Metals Research Association, for which we are indebted to Mr. G. L. Bailey, deputy director of this Association; the second deals with the work of the Iron and Steel Industrial Research Council of the British Iron and Steel Federation, for which we are indebted to Dr. W. H. Hatfield, chairman of the Council, for permission to abstract essential information from his lecture* dealing with research in this industry. These are typical examples of the work and organisation of research bodies that function for individual industries. Other associations in this group which are concerned with different sections of the metallurgical industries, as the British Cast-Iron Research Association, which, as pointed out in our issue of May last, has recently occupied new and commodious premises at Alvechurch, near Birmingham, and the British Refractories Research Association. These two latter organisations function on somewhat similar lines as those of the British Non-Ferrous Metals Research Association. Firms not yet connected with the Association serving their interests are strongly advised to make early contact and thus assist in increasing the scope of their activities to meet post-war demands.

* "Research in the Iron and Steel Industry," by Dr. W. H. Hatfield, F.R.S. The tenth Gluckstein Memorial Lecture delivered before the Institute of Chemistry, 1938.

The British Non-Ferrous Metals Research Association

By G. L. Bailey, M.Sc.*

THE position which the research associations have established for themselves since their inception at the end of the last war is largely due to the type of work which they have made their own especial province, work on the whole less academic in nature than that usually undertaken by the universities and technical colleges, yet broader in its scope than is generally possible in most works laboratories. Research associations, by pooling the subscriptions of a large number of manufacturers, are able to operate on a bigger scale than any but the largest industrial research laboratories. Moreover, not being responsible for day to day production, they are able to undertake relatively long-term investigations of problems of basic importance to the industry they represent. Although able to give immediate assistance to the smaller manufacturer with restricted facilities for technical control, the associations do not replace the works laboratories but supplement their activities by work in wider fields. The technical staffs of research associations understand and maintain close touch with the processes and requirements of industry, and at the same time keep fully abreast of advances in the more fundamental scientific research work. Machinery thus exists whereby the latest results of research and technical development in all parts of the world, and opportunities for experimental work without

interference with normal production, are at all times available to industry for dealing with its own problems.

This article presents a short account of the way in which the B.N.F.M.R.A. performs these services for the non-ferrous metals industry; but before doing so it will be as well, for the benefit of those who are not familiar therewith, to say a word or two about the Association itself.

The B.N.F.M.R.A. is a national organisation of producers, manufacturers and users of non-ferrous metals from whom it draws its income in the form of annual subscriptions. These are augmented by a substantial grant from the Department of Scientific and Industrial Research, by which the research associations were originally sponsored under "The Government Scheme for Industrial Research" of 1917.¹ To-day there are just over 340 companies who are members of the Association, which has a total income at present of about £43,000 per annum. In return for their relatively small subscription members enjoy an exclusive right to the results of the various investigations that are carried out and to the additional services, to be mentioned later, which the Association provides.

* Deputy Director, B.N.F.M.R.A.

¹ H. Moore, "Co-operative Research in the Metal Industries," *J. Inst. Metals*, 1943, L.XIX (3), pp. 93-108.

The Association is governed by a Council composed of representatives from its members and from certain Government and Service departments. Similarly constituted is the Main Research Committee, through which the Council surveys and guides the general course of the Association's research work carried out under the direction of the Director of the Association. A number of sub-committees, responsible to the Main Research Committee, are appointed to review from time to time the progress of individual investigations, and on these sit representatives of those sections of the industry that are interested in those particular researches. The work is thus at all times capable of being suitably and quickly modified as may be required by the changing needs of industry.

The work of the Association is described under three departmental headings: Research, Library and Information, and Development. No attempt is made to present a complete picture of the work in hand, or to give results of individual investigations, but the discussion of the method of working and the nature of the services offered to members is illustrated by reference to certain specific researches.

Research

In the past 20 years the research department has investigated a very wide range of problems. These include work on the production of sound castings in non-ferrous metals and alloys, corrosion and its prevention, particularly in marine service and in domestic water systems, spectrographic and other methods of metallurgical analysis, mechanical properties, including fatigue and creep resistance at room and elevated temperatures, and the production of new alloys with improved properties for specific applications. With the war have come new investigations on such matters as the use of secondary metals and new types of raw materials, and the development of alloys designed to eliminate or greatly reduce the use of metals in short supply. The greater part of the Association's researches are carried out in its own laboratories, but some work, e.g., that on the electro-deposition and surface treatment of metals, is carried on extramurally while the Association also contributes financially to certain external research work, such as the rolling and deep drawing of metals.

The work on the production of brass ingots for strip rolling may be taken as an example of the Association's methods of conducting an investigation into an industrial problem with the close co-operation of the industry concerned. At the time when this research was put in hand the operation of brass casting, particularly into the difficult shape of a relatively thin slab or strip ingot, had not been studied systematically. Existing technique had been built up almost entirely by empirical means, and few attempts had been made to bring the operation under scientific control. A complete review of the casting process was first required, and to that end a series of visits was made to representative works to observe the methods then in regular use. To this preliminary step was added that of thoroughly examining at various stages samples of rolled strip made from ingots cast in the works that were visited, studying their typical defects, and endeavouring to trace the connection of these with defects in the original ingots. On the completion of this survey a plan of research was decided upon.

A detailed account of the course of the investigation would be out of place here; for this, the reader is referred to the Association's Research Monograph No. 3, "The Casting of Brass Ingots," by R. Genders and G. L. Bailey. Broadly speaking, the experimental work consisted of an examination of the various factors which influence the soundness and other qualities of the ingot, such as the casting temperature and rate of pouring, the shape and material of the mould, the mould dressing, the dimensions of the ingot and the methods of feeding it. As the result of this the Association was able to suggest means of improving the existing procedure and also to recommend new and improved methods of casting. These suggestions were given practical effect by means of full-scale tests in members' works, and it is not too much to say that the results of this investigation have had a profound influence on brass ingot casting technique as practised at the present time.

The Association subsequently turned its attention to the sand and chill casting of other metals and alloys, particularly of light alloys. The production of sound sand castings of aluminium base and of magnesium base alloys have been fully studied, and similar work on phosphor bronzes and gunmetals is approaching completion. Work on the chill casting of light alloys has led to a full understanding of the occurrence of the troublesome "inverse segregation." In all these researches particular attention has been paid to reactions leading to the evolution of gases during solidification, and to the distribution and "feeding" of shrinkage unsoundness. Throughout these investigations close contact has again been maintained with the appropriate sections of the industry, and the experimental work has thus been guided throughout along lines likely to give the most valuable results. At the same time the investigators have been able to review the whole question of unsoundness in metals on solidification, and to make useful contributions to our basic knowledge on this vital subject.

It is appropriate to mention here that useful inventions arising from its work are patented by the Association for the protection and benefit of its members.

The Association has for many years taken a leading part in corrosion researches, and the classical work of Dr. W. H. J. Vernon² and Dr. J. C. Hudson³ on the resistance to atmospheric corrosion of non-ferrous metals was carried out under its auspices. More recently (in 1930) the Association took over the Institute of Metals Corrosion Research, devoted since 1910 to an investigation of the corrosion of condenser tubes. The value of the aluminium-brass condenser tubes patented by the Association has been clearly demonstrated in service, and much work has also been done on the development of cupronickel tubes for use under severe operating conditions. Apparatus devised in the course of these researches makes it possible to assess the value of new alloys for condenser systems by means of short time tests. For obvious reasons little can be said at present about more recent work in this field.

In the years before the war the corrosion troubles experienced in domestic water systems were a subject of investigation by the Association's corrosion experts

² W. H. J. Vernon. "Second Experimental Report to the Atmospheric Corrosion Research Committee (British Non-Ferrous Metals Research Association)." *Trans. Faraday Soc.*, 1927, **23**, 113-204.

³ J. C. Hudson. "Atmospheric Corrosion of Metals. Third (Experimental) Report to the Atmospheric Corrosion Research Committee (British Non-Ferrous Metals Research Association)." *Trans. Faraday Soc.*, 1929, **25**, 177-252, 474-496.

not only in the laboratory but by the examination of a large number of service failures in houses and commercial buildings. Much knowledge has been gained on the resistance to corrosion of galvanised iron tanks and cylinders, and of service pipes in galvanised iron, lead and copper. A Paper⁴ based on results of this work was read before the Institute of Heating and Ventilating Engineers in 1940 by L. Kenworthy.

The three B.N.F. ternary alloys of lead in which small quantities of cadmium and tin or antimony are added to the metal are examples of the development by the Association of new materials for a particular purpose. These alloys resulted from an investigation of the failure by intercrystalline cracking of pure lead cable sheathing when subjected to vibration, and were designed to overcome the trouble by providing greater resistance to fatigue. Suitable alloys were recommended, not only to replace pure lead in cable sheathing but also for water pipes and roofing sheet.

Another side of the Association's work is the examination and appraisal of new methods of metallurgical analysis and the development of special techniques for use with particular alloys and groups of alloys. Work on metallurgical analysis by means of the spectrograph was started nearly 20 years ago, and in 1933 the Association's Research Monograph No. 2, "Metallurgical Analysis by the Spectrograph," by D. M. Smith, was published. Since then the work has been greatly extended, particularly in the light alloy field, and the spectrographic method has become standard in some sections of the industry for certain purposes.

The results of an early research by the Association on the properties of soft solders are summarised in a book, "Tin Solders," by S. J. Nightingale, recently revised in a new edition by Dr. O. F. Hudson. While this may be regarded as the standard work on the tin-lead solders of pre-war days, the changed situation with regard to tin supplies has brought to the fore the question of substituting low tin or tin-free solders for the older types. The Association is taking a leading part in research on this problem, and is also organising through the Solders Advisory Panel an advisory service to help users in effecting the maximum economy of tin in soft solders.

The foregoing account of some of the Association's researches is not intended to be exhaustive. Actually more than 70 full-scale investigations, either completed or still in progress, have been put in hand since the Association was founded. Little has been said about the war-time activities of the Association, involving a considerable amount of special work for Government departments and a large extension of work on light alloys. In the latter field casting and welding have received special attention, and the work has dealt both with high-grade alloys and with the economic utilisation of secondary materials. The researches that have been more fully described, however, will serve to indicate the character and scope of the Research Department's work.

Development

As will be evident from what has been said, the Association's work does not end with the successful conclusion of laboratory experiments; the results have to be applied industrially if a return for the time and

money spent on them is to be secured. It is the particular task of the Development Department to assist the industry in this difficult final stage in the progress of a research. In doing so it calls freely upon the services of the appropriate investigators from the Research Department, and together they co-operate closely with the member, or members, in whose works the new methods are to be tried.

There is another way in which the Development Department brings the results of research carried out in the Association's laboratories and elsewhere to the aid of industry. The Association receives each year a large number of requests from members and Government departments for immediate assistance in technical problems arising in the course of their daily work. These technical enquiries, as they are called, are attended to by the Development Department with the assistance of the Research and Information Departments, each enquiry being treated in strict confidence. Some are quickly answered on available information, while others require experimental work to be done before an answer can be given. Wherever desirable, visits are made to members' works to discuss difficulties on the spot. This service is appreciated by members, but it has an additional value to the Association in that the Development Department is well placed for observing at an early stage the emergence of new problems affecting the industry as a whole or in part.

Library and Information

Of the greatest value to its own staff, and to its members, is the Association's library of metallurgical and related subjects. It contains some 5,000 books and more than 20,000 pamphlets, reprints, etc., all suitably catalogued and indexed. It is freely at the disposal of the staffs of member-companies, through a well-organised loans service.

A great deal of fresh material is constantly being added to the Library, nearly 200 different journals being regularly received and many others surveyed in other libraries. The important papers published are brought to the attention of members at an early date in the *Monthly Bulletin*, which is prepared by the Information Department, and in which short notes on new published literature and on the Association's own reports are presented.

The many contacts which the Information Department has with other libraries and with scientific and technical bodies, at home and abroad, greatly increases its usefulness to members of the Association. The Information Department also provides data from published literature on specific questions raised by members and prepares and distributes translations and reproductions of important foreign papers.

* * *

It is hoped that the foregoing account will have conveyed to the reader some idea of the work of the Non-Ferrous Metals Industry's Research Association. While the intention has been to present a broad picture of this work, it is not claimed that the B.N.F.M.R.A. has yet developed to the full its potentialities for service to the large group of industries it represents. The B.N.F.M.R.A., in common with the other research associations, has grown from small beginnings, and it has tackled first those problems which seemed at the time to be the most important and which were most amenable to investigation with the facilities available. As the

⁴ L. Kenworthy, "Corrosion Troubles in Heating and Hot-Water Systems," *J. Inst. Heating and Ventilating Engineers*, 1940, 5 (85), pp. 15-35.

Association has grown so has the scope of its work, but it cannot yet be said to have approached its full development. It is, however, becoming widely recognised that if Great Britain is to secure a leading position in

post-war markets, her industry must be technically pre-eminent. It is believed that the research associations, greatly augmented in income, equipment and personnel, will play an important part in achieving this end.

Activities of the Iron and Steel Industrial Research Council

THE creation of the Department of Scientific and Industrial Research in 1915, as a Government department was a stroke of genius. To place large sums of money to be expended in the encouragement of scientific and industrial research under an enlightened and sympathetic administration constituted a policy which has justified itself a hundredfold. It can be said that the scheme of collective research in operation in the iron and steel industry is indebted in part to the funds placed at its disposal by the Department of Scientific and Industrial Research. Without that substantial annual nucleus the industry would not have been able to create successfully its collective research system at so early a stage.

In considering collective research in the iron and steel industry, the problem to be solved was that of constituent competitive companies still collaborating for their general welfare. Under the aegis of the Iron and Steel Industrial Research Council, which is recognised as the Research Association of the industry, a system has been developed which is facilitating the increase of that basic scientific and technical knowledge necessary to the industry. The Council seeks to achieve this result through a number of main committees, which, together with their attendant sub-committees and panels, cover the major needs of the industry. It has been successful in enlisting for this purpose the services of a large number of the best scientific and technical men in the industry, or associated with its interests in the Services, the universities and scientific institutions. It has, in fact, mobilised the scientific personnel of the country, who can contribute to the solution of its problems.

Through the various main committees, their sub-committees and panels, the Research Council has organised scientific and technical inquiry in the following fields:—

1. Blast-furnace practice (including coke, ore, slag).
2. Open-hearth practice (including gas producers, instruments).
3. Heterogeneity of steel ingots (including ingot moulds, pyrometry).
4. Rolling mill practice (including roll design, furnaces, instruments).
5. Steel sheets.
6. Alloy steels (including thermal treatment).
7. Steel castings (including moulding materials).
8. Refractories.
9. Corrosion (including protective coatings).
10. Smoke abatement.
11. Collateral researches on allied subjects.

The Technical Department of the Federation and the Iron and Steel Institute service the various committees.

At the present stage in the development of the work of the Research Council, some of its committees have been established for some time and have been well tried by experience. Other committees of more recent formation have not yet had so much experience, but there is every reason to expect they will become equally effective. It will be recognised that the subject of collective research and mutual assistance has many difficulties, and every credit should be given to the many individuals who have assisted in overcoming these difficulties and who have helped in creating the flexible and indigenous system which has been built up under the Research Council.

The Research Council, in addition to their control of general policy, are also responsible in principle for the control of the details and supervision of all collective research work. It will be appreciated that, with the expansion in the amount of research work in progress, the supervision of the details by the Council has rendered it necessary that all proposals for researches are submitted to a Programme and Finance Committee, who arrange a programme of work for submission to the Council for final approval.

Research work carried on by individuals or conducted at works and laboratories, which is not reported to the Iron and Steel Industrial Research Council, is outside the activities of the Research Council. It should be appreciated that such independent research forms, and must continue to form, a large and preponderant proportion of the total research activities of the industry, only certain spheres of research being suitable for common action under an organisation representing the industry as a whole.

It will be appreciated that, whilst some constituent firms make substantial provision for research and technical development within their own organisations, other firms make such provision only in varying degrees. The attitude to the Research Council, for instance, as

We have just heard with profound sorrow of the death of Dr. W. H. Hatfield, technical research director of Messrs. Thos. Firth and John Brown, Ltd. He passed away in Sheffield just a few hours before he was to have taken part in a B.B.C. broadcast entitled "Made in Sheffield." The news will have come as a great shock to his many friends, as it has done to us, and particularly to his relatives, to whom we express sincere sympathy.

The unfortunate news was received when this issue was on the press, and an appreciation of his services to the iron and steel industry will be published in our next issue.

regards the nature of the service required varies, therefore, with the character of the personnel with whom it is working in particular constituent firms. The following question has necessarily to be dealt with—namely, "How far can a collective research organisation cater for the technical and scientific requirements of the different constituent firms, which, in the best interests of the industry, must still desire to preserve the stimulus of competition?" Such competition as regards technical efficiency, and also in technical and commercial enterprise is obviously necessary for progress, and it was decided that the work paid for collectively should be restricted to:—(a) Scientific and technical research which will be a benefit to the industry as a whole, and be likely to enhance its technical efficiency. (b) The encouragement and financing of research in extension of fundamental knowledge likely to be of ultimate value in advantageously affecting the processes and products of the industry.

It is true that research cannot be done by committees; it must be executed by individuals. But committees are a necessary means of organising research and of bringing together forces which make the handling of general problems possible; they can make available the means of conducting research, and can focus independent research activities into a common channel. Important functions for which committees are often better equipped than individuals are the planning where necessary of programmes which, if on a large scale, may be beyond the capacity of individuals, or individual firms; the publication of results and the facilitation, so far as they can assist, of their application in practice.

The research committees of the Iron and Steel Industry are responsible to the Iron and Steel Industrial Research Council; and are organised for secretarial purposes partly through the Technical Department of the British Iron and Steel Federation, and partly through the Iron and Steel Institute.

Research might be divided into that which is directed at improvement in the technique of production and explores the nature of its processes, and that which investigates the nature of products and is directed towards their improvement. When the committees organised by the Technical Department of the Federation and the Iron and Steel Institute respectively are considered, it will be found, but not without exception, that the Technical Department deals essentially with process committees, and the Iron and Steel Institute more with products. Sometimes the most useful work of the more practical committees is of a highly scientific character whilst the results of the researches conducted by the more scientific committees become practical as soon as they are applied.

Under this system, existing laboratories, whether associated with universities, technical institutions or works are encouraged to undertake particular lines of research or technical experiment. The result is found to be satisfactory and it gives encouragement to research in many quarters. It is easy by these means to draw on the facilities provided by the Government at the National Physical Laboratory, by the universities and in the research laboratories of the Services; while the encouragement of research stimulates the wider interests of universities and technical colleges and ensures growing interest in the problems in wider circles among investigators of all ages.

In general, the cost of the activities of the Research

Council, though considerable, is small compared with the resources of the industry or the benefits that may be attained by it. It is naturally incumbent upon the Research Council to ensure that funds are advantageously spent, but it is necessary at times that research shall be encouraged which is not calculated to yield results which can be used in practice in the immediate future. A proper division of research activities is attempted and the effort is calculated in part to be of immediate application, in part to be likely to be applicable as influencing the nature of the products, and in part with the object of increasing knowledge, even if there is no certainty of such knowledge being applicable within the near future. The view is held that a great industry should be prepared to incur a certain amount of expenditure for the advancement of knowledge in the belief that increase of knowledge will be of ultimate service.

One of the very great values of the committees has been the opportunity which such meetings give to scientific and technical officials of meeting one another and so facilitating the development of the spirit of exchange of experience and an interchange of views on many problems of collective interest.

It is perhaps interesting to consider in what way the general system working under the Iron and Steel Research Council differs from some of the other Research Associations. The activities cover a wider range than the ordinary Research Association. It has no research laboratories, and it has no director. Its work is carried out as already explained in various places and forms. It is rather in the autonomy of its major committees under the co-ordination of the Research Council that the solution is found. The building-up of group committees comprised of men specially interested and qualified to co-operate in the particular interest covered by the group has, in the main, been achieved. The function of the Research Council is to control the general policy of collective research within the Federation, but it leaves the individual research committees the utmost possible freedom to decide upon the scope and character of the researches undertaken. The chief aim is to promote and encourage research work in as many different centres as possible, rather than to set up hard and fast centralised schemes of collective research. It is, of course, clear that the steel industry of South Wales, for example, is of a different character from that of Sheffield, or the North-East Coast, and that the peculiar problems of these typical districts are best understood by trained minds actually engaged in the said districts themselves.

There are clearly alternative methods of collective research—i.e., either:—

- (1) By means of committees voluntarily set up within the framework of the Research Council organisation by the scientific and technical representatives of individual firms in the industry; or
- (2) By the formation of a central research body with laboratories acting for the whole of the industry.

The difference between these two methods is interesting, but it will be conceded that if research work of a particular industry is over-centralised there is more tendency for one group of ideas to dominate the character of the researches undertaken, whereas in method (1), which has so far been adopted, there is a greater possibility of a real exchange of ideas on fundamental matters between research workers of similar standing and of widely different points of view. It will be gathered, therefore, that the Council favour the maximum possible

freedom being given to individual research in the steel industry, and are opposed to undue bureaucratic control.

With regard to the researches of a more purely scientific character undertaken by the universities and the National Physical Laboratory, the Research Council seeks to co-operate, and frequently provides finance; such researches are discussed fully with the appropriate committees where they have originated, and then the utmost possible freedom is given to those engaged in this purely scientific work. The whole question of collective research is obviously a very difficult one, and its success or failure must necessarily depend almost entirely upon the outlook of the individuals taking part in it.

It is one of the axioms of our Research Council to stimulate the research spirit (in its widest sense) in all districts of the country and generally to raise the standard of manufacture in the iron and steel industry. This should be stressed because it is one of the problems accepted by the Federation and charged to the Research Council, and because this explanation has been given from time to time in justification of the formation of certain committees. In other words, committees must at times, at least in the early years of their formation, be regarded as being largely of an educational character as distinct from a true research character.

The only way to take full advantage of the results of collective research is for the individual firm to have a competent metallurgical staff capable of understanding and applying the results of such research. The best way to ensure the absorption into an industry of an adequate number of trained men from the universities, is to encourage each firm, however small, to employ a scientific staff, the size of which is naturally governed by the needs of the firm in question.

It is quite obvious to those who have studied the scientific side of steel manufacture, that in order to be

of any real service to a particular firm, the results of collective research need to be translated into some form which is applicable to the available plant, and to the existing or potential conditions. This can only be done by men who are fully acquainted with the working of the plant in question, and a mere mass of information of a scientific character broadcast to the industry at large can never take the place of a careful study and application of the results of research by men in daily contact with problems of any particular plant.

The essence of research is that it shall result in additions to knowledge; it is necessary that those who wish to profit by it shall be fully acquainted with the previous knowledge on the subject. If there are some who cannot follow the results of new work this is rather an admission of their inability to understand, and cannot be an acceptable criticism of the value of the research. In practical application this means that certain companies with inadequate research organisations and scientific assistance may wish, but are unable, to obtain the full benefit of such work, whereas other companies which maintain adequate organisations are able to take full advantage. It would be unwise to ignore the trouble and expense incurred by some firms in equipping themselves for carrying out and applying the results of research. Research must necessarily be an activity within an industry and cannot really constitute a service rendered to it, except as regards the extension of the general knowledge which is basic to the particular field.

The work carried out under a research council should rightly lie in the direction of tilling the ground so that exceptional effort of the individuals is made more readily possible, and in the long run the council's achievement in fostering a great interest in the background of knowledge is a reasonably satisfactory end.

The Iron and Steel Institute Annual Autumn Meeting

THE autumn meeting of the above Institute was held on October 14, 1943, at its offices in Grosvenor Gardens, London, S.W. 1; the President, Mr. James Henderson, being in the chair. Following the acceptance of the minutes of the previous meeting, which were taken as read, the President referred to the loss sustained by the Institute and by the iron and steel industry by the death of Sir Robert Hilton. "Within the last few days," he said, "our industry has sustained a very serious loss by the death of Sir Robert Hilton, which took place last Sunday (October 10), after a not very long illness. I am happy to feel that almost up to the last he was able and willing, as always, to do all he could to forward the interests of the iron and steel industry."

"I do not propose to give here a sketch of his career, which is known to many of you. From early years he was marked out for promotion, and the later years of his life were spent as managing director and, finally, as vice-chairman of the United Steel Companies. I was privileged to serve under him, so that I can speak at first hand of the great qualities he possessed, and which he used to the fullest extent in administration and organisation; he was certainly a great organiser. Not only did he leave his mark on the companies he served and on the industry, but he was a warm supporter of the

scientific side of the industry, and was very helpful to us in the Institute. He supported research both in his own companies and in the industry. We have lost a good friend and a great captain of industry—none deserved that title better than he did. I am sure you would wish to record our sympathy with his widow and family."

The members stood in silence as a tribute of respect.

President-Elect for 1944

The President announced that the Council had decided to nominate Mr. Arthur Dorman to succeed him as President of the Institute. "Apart from his association with Messrs. Dorman Long, and many other companies," said Mr. Henderson, "he is a vice-president of the Federation of British Industries, past-president of the British Iron and Steel Federation, and of the National Confederation of Employers' Organisations, and has been a member of this Institute for many years. I do not think we could wish for a more experienced and distinguished occupant of this chair. He is still young enough to be full of vigour and fresh ideas."

In thanking the Council and the Institute for the manner in which his nomination was received, Mr. Dorman expressed himself as very modest in accepting because he regarded his technical qualifications as meagre

"We have to remember," he said, "that the world to-day is very research-minded, and that there are great possibilities in front of this Institute. When one looks back at the distinguished men who have filled the office of President, it must make anyone who is not specially endowed with technical knowledge a little nervous about occupying it. I can, however, promise that I will do my best to maintain the reputation of the Iron and Steel Institute, and I hope I shall not fail in achieving that aim."

Retiring Officers

It was announced that the following officers would retire in rotation at the Annual General Meeting in 1944:—

Vice-Presidents.—Dr. W. H. Hatfield, F.R.S., Sir William J. Larke, K.B.E., and Mr. C. E. Lloyd.

Members of Council.—Principal C. E. Edwards, F.R.S., Mr. C. H. Latham, Mr. E. F. Law, Mr. R. Mather, Sir Arthur Winder, and Mr. N. H. Rollason.

It was also announced that Mr. Bernard Thomas, president of the Staffordshire Iron and Steel Institute, had succeeded Mr. Albert Wright, and Provost J. Tennent, president of the West of Scotland Iron and Steel Institute, had succeeded Col. Alan Stein, as honorary members of Council.

Legacy from Sir Robert Hadfield

It was announced that the late Sir Robert Hadfield left a legacy of £250 to the Institute; its utilisation is left to the discretion of the Council. At present it is difficult to decide the purpose for which this money may be applied, it has, therefore, been invested and its use will be considered in a year or two's time. The President took the opportunity of expressing the appreciation of members that Sir Robert Hadfield remembered the Institute, which, in his lifetime, he had helped so much by his work.

Andrew Carnegie Research Grant

It was announced that the Council had awarded a Carnegie Scholarship grant to M. S. Wang, of Sheffield University. The amount of £150 is awarded to assist in a research on the effect of repeated heat-treatment on steels.

Bessemer Gold Medallist, 1943

At the annual meeting of this Institute held last May, Mr. J. H. Whiteley was awarded the Bessemer Medal, and when calling upon him to present his paper on "A Study of Austenitic Grain Growth in Medium-Carbon Steels," the President took the opportunity of referring to the fact, and of again congratulating him and wishing him long life and health.

In his reply, Mr. Whiteley apologised for his absence from the last meeting; had he known what was to happen he would have made strenuous efforts to be present. "On many occasions," said Mr. Whiteley, "I have listened to the replies of those who have received this honour, and each time I had the comfortable feeling that such an ordeal would never be mine."

Mr. Whiteley gave one or two incidents from his life, and expressed a wish to name three gentlemen in connection with this award. The first is Mr. Arthur Braithwaite, with whom he became assistant at the age of 22. The next, Dr. Stead, for his example and inspiration and encouragement given, which has been invaluable. "At the last meeting," said Mr. Whiteley, "the President

referred to a paper¹ written jointly by Dr. Hallimond and myself, entitled 'Acid Hearth and Slag.' Whatever excellence that paper may have half at least is due to Dr. Hallimond. He was with me for four years during the last war, and it was to me an invaluable time."

Mr. Whiteley thanked the President, the Council, and the members of the Institute for the great honour conferred upon him.

Technical Sessions

In addition to the paper by Mr. J. H. Whiteley on "A Study of Austenitic Grain Growth in Medium-Carbon Steels," several other papers were presented for discussion. These included "The Structure and Segregation of Two Ingots of Ingot Iron, one Containing Lead," by Dr. L. Northcott and Mr. D. McLean, and "Leaded Manganese-Molybdenum Steel," by Dr. T. Swinden, which were considered jointly; "The Solidification and Cooling of Steel Ingots, Notes on an Examination of Three Typical Ingots," by Mr. E. F. Law and Mr. V. Harbord, and "The Physical Chemistry of Open-hearth Slags," by Dr. J. White.

On page 287 is an abstract from Mr. Whiteley's paper; reference will be made to the papers on lead-bearing steels in a later issue.

¹ J. H. Whiteley and A. H. Hallimond, "The Acid Hearth and Slag," *Jour. I. & S. Inst.*, 1919, No. 1, p. 199.

Surface Structures and Chemistry of Heat-Treated 18-4-1 Steel*

THE effects of different forms of heat-treatment on the composition, microstructure, scale formation and hardness of the steel were studied. Micrographs show the characteristic structures of the steel after annealing, after oil-hardening, and after oil-hardening and tempering. Specimens quenched in oil after heating in a blacksmith's forge were partly carburised and decarburised. Analyses of the surface material of specimens before and after preheating at 843° C. in reducing, neutral and oxidising atmosphere showed that the original carbon content of 0.72-0.74% remained practically unchanged. The results of tests are presented in which specimens were heated to 1,260-1,343° C. in reducing and in oxidising atmosphere ranging from CO 15.8 and O 9.3% after preheating in a reducing atmosphere, and the effect of temperature, holding time and atmosphere composition are discussed. The carburisation and decarburisation of specimens after treatment in reducing and oxidising atmosphere are examined and the carburising action of two types of molten salt baths is explained. The general conclusions reached are:—

(a) When the steel is heated for hardening a metal oxide zone is always formed regardless of the method of heating, and this zone is at a minimum when an atmosphere containing 8-12% of CO is used.

(b) Neither carburisation nor decarburisation occurs during preheating at 816°-71° C.

(c) The steel may be held at high temperature for 10 mins. in a reducing atmosphere without decarburisation occurring.

(d) The behaviour of the carburised area during tempering depends on the degree of carburisation and the proportion of retained austenite; the latter can be decomposed to form martensite by re-tempering.

* W. A. Schlegel, *Iron Age*, 29, 45-50, 30, 48-49, 31, 54-8.

A Study of Austenitic Grain Growth in Medium-Carbon Steels*

By J. H. Whiteley, F.I.C.

Eight interdependent factors were found to influence grain growth, viz.: Temperature, length and rate of heating; initial grain size, hot-work and the aluminium content of the steel; the rate of cooling from the solidus to 1,200 °C., and the state of the carbides. With so many possible combinations an exhaustive examination was impracticable, but the results of this careful work, from which this abstract is taken, constitutes an important contribution to the subject.

DURING the last decade or two grain growth within the completely austenitic range has been a prominent subject of investigation. In America especially, both the control of the grain size and its influence on various physical properties have been intensely studied by metallurgists, and to them much credit is due for the considerable advance which has accrued. The fact that a stipulated grain size is now not uncommonly included in specifications is an indication of the progress achieved. Yet, notwithstanding the large amount of work which had been done, much remained obscure when the present inquiry began about seven years ago. Little was known with any degree of certainty as to the factors governing grain development in unstrained steels except that the addition of small amounts of alloys such as aluminium or vanadium retarded growth, and, even yet, no complete agreement has been reached on their mode of operation. The explanation most in vogue is that oxygen is the primary cause. It is asserted that when the steel at tapping contains an appreciable quantity of that impurity, extremely fine oxide particles of the added elements are formed and dispersed throughout the metal. These then serve both as nucleation centres and as inhibitors of grain growth. The evidence in support of this theory was well summarised some years ago by Bain.¹ Although the arguments that he advanced are impressive, it is nevertheless difficult to see how fine inclusions can bring about many of the growth effects which are described in the eight sections of the present paper. Taken as a whole, the results suggest rather that the state and character of the carbide as the critical range is entered are among the main determining factors.

In the first section the author makes various introductory remarks primarily concerned with details of the investigation of austenitic grain growth, and states that in the study of this subject a trustworthy means of revealing the grain size existing at the temperature of heating is an essential requirement. Fortunately, several methods are now available, details of which have been published at various times. An excellent summary by Ward and Dorn² may be mentioned. One of the latest procedures to be devised is that of heat-etching in an inert atmosphere.³ While each of the methods has some specific advantage, none is without a drawback of one

kind or another. Even the well-known carburising test has limitations. It is of little use at temperatures below 900° C., because a cementite network cannot there be readily produced, owing to the slow rate of carbon absorption. For the same reason the method is not suitable for the study of initial grain growth at any temperature. Moreover, the assumption is made, no doubt correctly in most cases, that the introduction of carbon causes no alteration in grain development. The alternative method of ferrite precipitation happens to be free from these particular disabilities. As in the carburising test, reliance is there placed on the disposition at the grain boundaries of the phase which separate as the steel approaches the Ar point, and since in medium-carbon steels that phase is ferrite, the method is applicable to them at all temperatures within the austenitic range, whether the heating periods be long or short. It thus appeared to meet the needs of the present investigation, and was accordingly adopted. In using the method one precaution has to be taken which is of less importance in the carburising test. To obtain satisfactory results it is necessary to regulate the rate of cooling so that only a fairly thin network is formed, for if too much ferrite is precipitated the deterioration of the smaller grains may be marred.

The subject of grain growth can seldom be adequately treated without the aid of photographic illustrations; but while these are unquestionably desirable, their number may rapidly become excessive, especially when the work is extensive. This difficulty is not insuperable, however, for many grain sizes can be recorded numerically, and in preparing this paper there was no option but to employ that method as much as possible. Each figure given in the tables denotes the average number of grains in a standard area of 10 sq. in. at a magnification of 100 diameters, so that by reference to the A.S.T.M. chart the size can readily be visualised.

Particulars of the steels on which much of the work was done are given in Table I. In every instance the sample was taken from a bloom or billet rolled from the lower half of the ingot. The majority of the steels were made in the acid open-hearth furnace under a fairly standard practice in which the FeO content of the slag was reduced to 19%, or less, at tapping by limestone additions during the boiling period. Several basic steel samples, other than those included in Table I, were also employed, but chiefly for checking the results obtained on the acid steels, and since the agreement was good no further mention is made of them. For the most part

* Paper No. 12/1943 of the Alloy Steels Research Committee, published by the Iron and Steel Institute. (Preprint, 56 pp.)

¹ Bain. *Journal of the Iron and Steel Institute*, 1938, No. 11, p. 339.

² Ward and Dorn. *Metals and Alloys*, 1939, vol. 10, No. 3, p. 74.

³ Day and Austin. *Transactions of the American Society for Metals*, 1940, vol. 28, p. 354.

TABLE I.—DESCRIPTION OF SAMPLES.

No.	Type of Steel.	Aluminium added per Ton, Oz.	Class.	Condition.	Analysis, %.					Slag Iron, %.
					Carbon.	Silicon.	Phosphorus.	Sulphur.	Manganese.	
1	Acid open-hearth	Nil	Coarse-grained	Billet	0.45	0.12	0.044	0.033	0.71	14.4
2	" "	Nil	"	"	0.45	0.14	0.033	0.045	0.72	14.8
3	" "	Nil	"	"	0.44	0.10	0.042	0.034	0.63	12.6
3A	" "	1	"	"	—	—	—	—	—	—
3B	" "	3	Mixed-grained	"	—	—	—	—	—	—
3C	" "	9	"	"	—	—	—	—	—	—
3D	" "	16	Fine-grained	"	—	—	—	—	—	—
3E	" "	32	"	"	—	—	—	—	—	—
4	Basic open-hearth	3	Coarse-grained	"	0.44	0.11	0.012	0.043	0.63	9.6
5	Acid open-hearth	16	Fine-grained	"	0.475	0.23	0.033	0.038	0.71	13.0
6	" "	16	"	"	0.42	0.21	0.027	0.037	0.63	12.7
7	" "	16	"	(Bloom Billet)	0.465	0.18	0.040	0.055	0.75	14.7
8	" "	16	"	(Billet)	0.44	0.20	0.037	0.038	0.65	14.1
9	" "	Nil	Coarse-grained	(Pit sample Billet)	0.48	0.11	0.035	0.048	0.80	13.1
10	Basic open-hearth	Nil	"	(Pit sample Billet)	0.45	0.24	0.036	0.031	0.88	8.3

the steels enumerated in Table I can be divided into two very important classes, according to their behaviour in the McQuaid-Ehn carburising test. Samples 1, 2, 3, 3A, 4, 9 and 10 contained little or no aluminium, and showed a grain size in the vicinity of No. 3, whereas a No. 7 size appeared in Nos. 3D, 3E, 5, 6, 7 and 8. Throughout the paper the terms coarse-grained and fine-grained are exclusively used to denote respectively these two classes.

When examining grain growths at temperatures up to 1,150° C., the specimens were heated in a horizontal electric furnace wound with Kanthal wire, so spaced that along one half a uniform temperature could be maintained while along the other half it steadily diminished to below 600° C. The gradient end was of advantage in several ways, as, for instance, in cases where the specimen was to be subjected to a rapid temperature change. Tests above 1,150° C. were usually carried out in a Silit-rod furnace. Except when the heating period to be given was very short, the specimens were placed in silica tubes 11 in. long, fused at one end, the other end being closed with clipped rubber tubing. Decarburisation to any appreciable extent was avoided either by putting in extra pieces or else by the addition of a small quantity of high-carbon steel drillings.

In dealing with the influence of the speed of heating on grain growth three widely separated rates were generally employed, and throughout the paper they are designated as slow, rapid, and very rapid. The precise meaning of these terms is as follows:—

"Slow heating" signifies that the temperature was raised through the critical range at from 6° to 8° C. per min., and when a rate is not mentioned this one is always implied.

"Rapid heating" denotes an average temperature rise of between 300° and 500° C. per min., to obtain which the tube containing the specimen was inserted in the furnace at, or near, the temperature of the test; in such tests specimens weighing less than 2 g. were taken.

"Very rapid heating" indicates an average temperature rise of about 800° C. per min. The specimen weighed approximately 0.29, and was roughly in the form of a cube. It was placed at the clipped end of the tube, and afterwards dropped quickly on to one or more slowly heated specimens as soon as the required temperature was reached.

Since rapid and very rapid heatings necessitated the use of small specimens, it was clearly of importance to

ascertain at the outset that grain growth was not affected by mass. Several tests were therefore carried out on both coarse- and fine-grained steels, in each of which an hermetically sealed 3-lb. block containing two specimens of the same steel, weighing 0.2 and 0.8 g., was heated at temperatures between 950° and 1,100° C. In no instance was there any observable difference between the grain sizes which developed in the specimens and in the block. Thus, the experimental evidence

demonstrated that mass of itself was not a factor concerned in growth, and consequently it received no further attention.

Although ferrite precipitation is frequently employed for the delineation of austenitic grains, the validity of the method seems to have been taken for granted. Certainly, with medium-carbon steels, it reveals sizes corresponding closely with those brought out by the carburising test, as the illustrations on the A.S.T.M. standard chart indicate. During the present investigation the two methods were compared a number of times both at 930° and at 1,000° C. Typical results obtained at the latter temperature are reproduced in Table II. In each instance the two specimens were heated together for 4 hours, the carburised one being then furnace-cooled and the other air-cooled. Although the agreement was always satisfactory, it nevertheless seemed desirable to establish the reliability of the ferrite-precipitation method in some other way. For that purpose recourse was had to a surface effect produced in sealing, which the author had studied some years ago.⁴

TABLE II.—GRAIN SIZES GIVEN BY CARBURISING AND FERRITE PRECIPITATION METHODS IN TESTS OF 4 HOURS AT 1,000° C.

Sample No.	Average Number of Grains in 10 sq. in. at 100 Magnification.	
	Carburised.	Ferrite Precipitation.
1	25	24
3, forged	45	45
6	804	796

The method is described as follows:—

When the steel is heated in air the boundaries of the surface grains become very resistant to attack by the usual acid reagents, so that they stand out in strong relief on the etched surface. With Le Chatelier's cupric reagent the contrast produced is even more marked. A ready means is thus afforded of locating the austenite grains which existed immediately beneath the scale at the temperature of heating, and in order to compare the network of structure thus revealed with that shown by ferrite precipitation it was merely necessary to bring the two into contiguity. There, however, a difficulty was encountered due to surface decarburisation. In the first attempts a narrow zone, in which ferrite largely predominated, separated the scaling network from that

⁴ Whiteley. *Journal of the Iron and Steel Institute*, 1935, No. 1, p. 181.

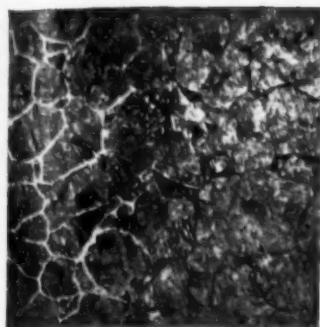


Fig. 1.—Scaling and ferrite grain boundaries. Cupric etch. $\times 90$.

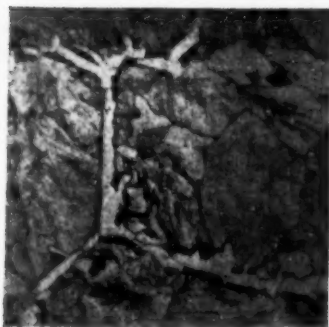


Fig. 2.—Ferrite at grain boundaries. Very slow etch. $\times 450$.

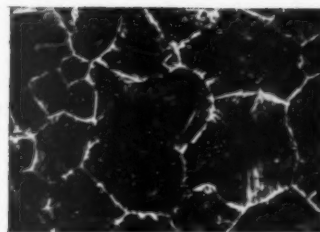


Fig. 3.—Sample No. 1, 27 hours at $1,020^{\circ}\text{C}$. $\times 55$.

formed within the steel. Eventually it was found that a much closer juxtaposition could be obtained by covering a part of the ground surface to be scaled with a thick layer of a stiff paste made of finely ground alumina mixed with 6% of sodium carbonate and water. When the paste had dried the specimen was inserted in the furnace at 970°C ., where it remained for 30 mins. before being air-cooled. On detaching the scale there was usually a slight step downwards from the covered area. The raised portion was levelled and the surface then prepared on emery papers for examination. In that way a junction of one network with the other was effected, and in the many specimens of different steels thus treated, the two were seen to be one and the same. At the dividing line no grain boundary was disclosed by scaling which was not also shown by ferrite precipitation. A typical example is reproduced in Fig. 1; there the scaling network is white, while the ferrite network has been darkened by copper deposition.

Although it is convenient to describe the thin layers of the precipitated phase, whether they be ferrite or cementite, as grain boundaries, the term is not strictly correct, for in all probability the material in any one layer was provided by both the adjacent grains, the original boundary being thus replaced by a band of new small crystals. In medium-carbon steel the granular formation of these marginal layers is but seldom brought out by ordinary etching methods, the narrow bands appearing to be continuous, especially in the case of cementite. It was found, however, that the grains in a ferrite network could be revealed by a very slow etching in a solution consisting of 95% alcohol containing 0.6 g. of picric acid per 100 c.c. A typical example is reproduced in Fig. 2, which shows the tabular crystals in a ferrite network resembling that illustrated in Fig. 3. In applying the reagent the quantity taken has to be regulated to suit the size of the specimen so that the initial rate of attack soon slackens. Results similar to that shown in Fig. 2 were obtained in the following way: The specimen, weighing about 0.80 g., was placed slantwise, with the prepared surface upwards in a crucible of 4 c.c. capacity, which was then filled with the reagent, covered and left undisturbed overnight. On removing the specimen the surface was cleaned by a light and repeated rubbing on a piece of moist Selvyt cloth until the structure was satisfactory under the microscope. A slight pitting occasionally took place, but it was chiefly confined to the edges.

This method of etching not only revealed the granular structure of the ferrite layers comprising the network, but also the sub-boundaries within the pearlite areas,

some of which are evident in Fig. 2. They are due to the fact that the transformation started at more than one point on the margins of the austenite grains, and as a result each pearlite grain contained several differently orientated areas. That mode of crystallisation occurs more frequently when the transition is fairly rapid, as was noted by Carpenter and Robertson⁵ in their study of the austenite-pearlite inversion.

From the foregoing, which comprises practically the whole of the first of the eight sections, each concerned with interdependent factors, influencing grain growth, it will be appreciated that the author has carefully studied the subject, and the results of his investigations constituted an important contribution to the literature on austenitic grain growth. The interdependent factors discussed include: Temperature, length and rate of heating; initial grain size, hot-work and the aluminium content of the steel; the rate of cooling from the solidus to $1,200^{\circ}\text{C}$., and the state of the carbide. The manganese content of the steel may be yet another factor.

With so many possible combinations an exhaustive examination of the subject was impracticable, but from the work done the conclusion may be drawn that any particular grain size formed by heat-treatment in a given medium-carbon steel is the result of the concerted action of these various factors. It is shown that grain refinement takes place almost entirely at the A_{c1} transformation, and is completed during the passage through the critical range. In the coarse-grained steels the rate of growth then steadily increased with the temperature of heating, and below about $1,100^{\circ}\text{C}$. a state of virtual stability was eventually reached. In the fine-grained steels a remarkable halt in grain development occurred almost immediately after the refinement. Below 900°C . this period of inactivity was indefinitely long, and, as a consequence, the steel was fine-grained in character. Above 900°C . the halt became progressively shorter, but at any one temperature its actual duration could be varied widely by changing the rate of heating, the initial grain size and other factors. When once the halt was passed mixed grain sizes began to appear, consisting of large dominant grains embedded in small ones. Dominant growth of a relatively few grains was an effect also observed in coarse-grained steels, but the small grains did not remain so inactive. As the temperature was raised the mixed grain sizes gradually disappeared. After refinement by forging, a smaller grain size grew in coarse-grained steels at temperatures up to at least $1,000^{\circ}\text{C}$. than after an equal refinement by

⁵ Carpenter and Robertson. *Journal of the Iron and Steel Institute*, 1932, No. 1, p. 319.

heat-treatment, and it is shown that the disparity was not due to differences between the banded heterogeneity or to the distribution of the inclusions. Hot-work seemed to be the cause. In fine-grained steels this factor was not so much in evidence, owing to the mixed grain sizes which were formed in the temperature range where it operated. The production of a coarse-grained steel was found to depend on the rate of cooling from either the liquid or solid condition to 1,200° C. Unless the rate was fairly slow the steel was fine-grained in character. In the absence of manganese the change to the coarse-grained type could not be induced even by a

very slow cooling. The state of the carbide had an important influence both on the extent of growth and on the length of the halt. Complete spheroidisation of the pearlite greatly minimised growth, especially in fine-grained steels. The effects of quenching and of quenching and tempering on growth were not the same as those of spheroidisation. Shortly after the first halt in well-tempered pieces with a large initial grain size a second half occurred which persisted for a long while to above 1,050° C. Attempts were made to convert solid coarse-grained steels into fine-grained by diffusing aluminium into them, but the results were inconclusive.

New Phosphate Coatings with Unusual Corrosion Resistance

By George Jernstedt

Phosphoric acid solution treatments of steel and zinc surfaces have long been used to increase paint adhesion, and thereby improve corrosion resistance. An investigation, by Jernstedt, of a pre-dip solution is outlined, and phosphate coatings employing this pre-dip are claimed to have greatly improved corrosion resistance and to have many applications where no supplementary organic coating is required.*

THE phosphating of certain metals for increased paint adherence has come into widespread use in the last few years. The first patents concerning this method of treating metal surfaces were issued to Coslett in 1908, and covered a solution of phosphoric acid and ferrous sulphate in definite proportions. A few years later Coslett¹ added zinc sulphate to the bath and obtained better results. In 1918 the Parker Company discovered that the character of the coating was improved by the use of a solution of primary manganese phosphate. This led to the process subsequently known as "Parkerising."

In the earlier processes using phosphoric acid, the time varied between one day to one week. The Parkerising process at first usually required a treatment of from 3 to 4 hours. In subsequent developments, efforts were concentrated on the reduction of time of processing. The addition of manganese dihydrogen phosphate produced an appreciable reduction in time of treatment, and somewhat later the addition of a small amount of a copper salt reduced the time to about 10 mins. This shortened time permitted the use of the process on a conveyor system. Still later improvements were directed towards further time reduction, as well as spray application, and improvement of the crystal structure of the surface of the basis metal. The addition of alkali nitrates, or nitrites, was an important step in the development of better phosphating solutions.² Probably the most generally used names of this type of surface treatment are "bonderising" and "Parkerising." All through the initial period of development it was evident that a finely grained crystalline steel surface was desired.

An unusual phenomenon observed in treating the metallic surfaces, such as zinc-plated steel, with phosphoric acid solutions is known as the "wiping effect,"

resulting from the wiping of the plated surface just prior to phosphating. By mechanically wiping the surface the structure of the resulting phosphate coating can be greatly refined and the corrosion resistance of the finished article decidedly increased. It has been found that the refinement of the structure of the surface is not due to any chemical action, but is due to the physical action of wiping the basis metal, notably zinc, with a cloth, brush, hand, etc.

Present commercial specifications for phosphate coatings always include a subsequent treatment because the film obtained has little, if any, value of its own. In the lacquering of zinc-plated steel the presence of corrosion is still evidenced by the white powder formed from the zinc, and by the subsequent flaking off of the organic finish. Steel parts used on the inside of instruments, relays, watt-hour metres, etc., require that there be not only no rust formation, but also that no other corrosion products are formed which might get into bearing surfaces and increase friction.

In an effort to find a solution to this problem phosphate and chromate treatments over zinc have been investigated. The wiping of surfaces was impractical from a production standpoint, due to different shapes and sizes of parts involved. At the time of starting the experiments a pre-dip compound was commercially available. This dip consisted of a 1-2% solution of Fixodine.³ This solution would produce a uniform fine grain in the subsequent coating, but required good rinsing after the pre-dipping because the oxalic acid poisoned the phosphate bath. It also attacked the zinc-base metals and required analysis and control. This solution was not further investigated.

It was decided to seek a satisfactory pre-dip solution which would simulate the effect of wiping the metallic surface and give a fine grain corrosion-resistant coating and a good bonding finish for lacquering. If possible,

* Paper presented at General Meeting of the Electrochemical Society, April, 1943. (Preprint, 53-40.)

1 T. W. Coslett, U.S. Pat. 1,007,069 (1911).

2 Tanner and Lodeesen, U.S. Pat. 1,911,726 (1933).

3 G. C. Romig, U.S. Pat. 2,164,042 (1939): A solution of oxalic acid.

the pre-dip to require little control or adjustment and be low in cost.

The pre-dip solutions investigated varied from that of strong mineral acids to weak organic acids; from that of strongly oxidising to strongly reducing solutions; from low pH to high pH solutions, in addition to various complex solutions.

Working on the theory that it was desirable to have absorbed on the metal surface a film of an anion or cation already present in the phosphating bath, a solution of disodium phosphate was tried. This particular solution gave an unusually fine grain and uniform appearing surface. Subsequent tests proved that the surface provided excellent paint adhesion and a corrosion resistance superior even to that of the phosphate coatings wiped previous to processing. However, it was not possible to locate another bottle of disodium phosphate with the same properties as this first one for quite some time.

The monosodium and trisodium phosphate varieties gave negative results. A little later several batches of satisfactory disodium phosphate were obtained. Purity did not seem to be a factor, because an active "commercial" salt and an active "reagent grade" of the same compound (Sorenson's) gave practically the same results. It was evident, though, that the anhydrous compounds were superior. The activity of a satisfactory disodium-phosphate pre-dip invariably deteriorated on standing. The rate of deterioration was accelerated by heating and decreased by cooling. These observations held good even after the bath had been kept in a darkened room or in a sealed container. The optimum concentration for a good disodium-phosphate-aqueous pre-dip was approximately 1-2% by weight on the dry basis.

The investigation continued along many lines, but eventually an entirely different solution of the problem was obtained. At one time a turbid solution gave good results, which was contrary to previous tests. By many filtering and concentrating operations a quantity of the turbid material, sufficient for a spectrographic determination, was separated and analysed. Although there were other impurities of greater concentration in the salt, it was the titanium, approximately 0.001%, in the disodium phosphate which proved to be the active agent. Small amounts of a titanium salt properly added to disodium phosphate will give an unusually active pre-dip solution which can be used in conjunction with many of the commercial phosphating baths.⁴ Such a process has been in continuous commercial production since 1941 with very gratifying results.

Titanium Pre-dip Process

If 1% solution of disodium phosphate is made up and a soluble titanium salt added to it the bath will be relatively inactive. It was found to be necessary to add the titanium salt in the proper concentrations to the original solution of disodium phosphate at the time it is made up. This provides for the evaporating to dryness of the disodium phosphate in the presence of the titanium ion. If this procedure is not followed the solution made up from the material will be relatively inactive. This explains why most of our originally discovered active salts were of the anhydrous variety.

The titanium-disodium-phosphate pre-dip remains active for weeks at a time, even under heavy production schedules in a conveyer system with no analysis or

control. The relative cost of the pre-dip is negligible, since the disodium-phosphate concentration is but 0.5-1%, and the titanium compound is made up to approximately 0.01% of the disodium phosphate.

There are other advantages secured from this pre-dip. The finish obtained either on steel or on zinc is unusually smooth and fine-grained. It is possible to apply a single coat of lacquer without any rub-down operation of the surface. The phosphate surface is also sufficiently smooth, so that when applied to either zinc or steel it is not necessary to apply any subsequent organic coating for any applications. The processing times of the commercially available phosphating processes were reduced to approximately one-third of the original. The temperature of processing also can be reduced appreciably. These factors increase the field of application for this type coating, especially from the standpoint of conveyer operations.

The Constitution of Magnesium-Manganese-Zinc-Aluminium Alloys in the Range 0.5% Magnesium, 0.2% Manganese, 0.8% Zinc

II.—The Constitution of the $MnAl_6$ Phase*

IN the quaternary alloys of aluminium with magnesium, manganese and zinc the phase usually called $MnAl_6$ occurs as a constituent of most alloys containing more than very small percentages of manganese. According to the X-ray work of Bradley and Jones¹ the phase has the composition $MnAl_7$, although Dix, Fink and Willey,² from the analysis of chemically extracted residues, concluded that the composition was $MnAl_6$, and this formula has been generally accepted. It is therefore of interest to examine the exact composition of this phase and the extent to which it can dissolve magnesium or zinc, because, if it can be established that the phase is of fixed composition, some of the complicated ternary and quaternary diagrams can be interpreted, or calculated, in a comparatively simple way. This paper, by Dr. G. V. Raynor and Dr. W. Hume-Rothery, describes some experiments made in this connection.

The most satisfactory way of approaching the problem would be to prepare alloys of different compositions, and to examine these microscopically after suitable annealing treatment. Unfortunately, this method is very difficult, if not impossible, owing to the extreme brittleness of the alloys, and to the fact that the form of the manganese-aluminium equilibrium diagram is such that marked segregation tends to occur during the solidification of an alloy of composition $MnAl_6$. The authors have therefore discarded this method, and have used instead methods based on the chemical analysis and X-ray examination of $MnAl_6$ crystals. These crystals were isolated by anodic attack from binary manganese-aluminium alloys, and from ternary and quaternary alloys containing magnesium and/or zinc.

Chemical analysis confirmed the composition $MnAl_6$, and showed that no magnesium was taken up by the

* *Jour. Inst. Metals*, 1943, Sept., pp. 415-421.

1 A. J. Bradley and P. Jones, *Phil. Mag.*, 1931, (vii), **12**, 1,137.

2 E. H. Dix, Jr., W. L. Fink and L. A. Willey, *Trans. Amer. Inst. Min. Met. Eng.*, 1933, **104**, 335.

4 Jerstedt, U.S. Pat. 2,310,239 (1943).

compound when this element was present in the alloy. This was confirmed by X-ray methods, which were used to obtain the interplanar spacings of different samples of $MnAl_3$ crystals. $MnAl_3$ crystals extracted from alloys containing zinc gave interplanar spacings identical with those of pure $MnAl_3$ crystals, but owing to the approximate equality of the atomic diameters of zinc and manganese, this is not a conclusive test for the absence of zinc. Chemical analyses suggested that small amounts of zinc are taken up into solid solution, but that they do not exceed 0.4% in crystals extracted from alloys in the range magnesium 0-5%, manganese 0-2%, and zinc 0-8%.

Manganese from the Gold Coast

THE deposits of manganese ore at Nsuta, in the Gold Coast, are located 39 miles by railway from the modern port of Takoradi, completed only in 1928, and were discovered by the late Sir Albert Kitson, then Director of the Geological Survey, at the very opportune date of 1914. Owing to the demand for manganese by the allies, prospecting and development were carried on concurrently, and even by 1916 4,275 tons were exported. Present production is of the order of 300,000 tons annually, and altogether the total output from this property, which virtually accounts for all Gold Coast production, has been over 7½ million tons.

At no time more than the present has manganese been so important, when probably more than 95% of the total world output is used in making plain carbon steel, to say nothing of its many other important uses in non-ferrous alloys, torch batteries, medicines and chemicals.

With the completion of Takoradi harbour the present operating company installed on the main quay an electrically operated ore-handling plant with an hourly capacity of 1,000 tons, so that a 10,000-ton cargo boat could be despatched in the course of a day. The United Nations, therefore, are extremely fortunate in possessing this major source of manganese situated in such a favourable position for exports. The Nsuta property is, in fact, the largest single producer of manganese in the world, though certain areas when considered collectively, such as the Chiatara district in the Caucasus, have a larger aggregate production.

The importance of these Gold Coast ore bodies, which occur in lenticular masses up to 1,000 ft. in length and up to 100 ft. in thickness, have long been under consideration by Sir Albert's successors in the Geological Survey of the Gold Coast, and their studies on the geology of these deposits, just recently published, has thrown an immense amount of light on the mode of occurrence both of the ores and of the associated formations, as well as on their probable origin. This type of information will, of course, be invaluable in developing the present and future ore bodies to be worked.

Besides describing the above features, Mr. Service's memoir* deals with the discovery and opening up of the Nsuta ore body, the type of country in which it occurs, as well as reviewing the previous literature on this

subject. The work is illustrated with photographs of the surrounding country, of the ores and associated rocks, and with photomicrographs of some of the two latter. Excellent plans of the area, geological maps and sections also accompany this memoir, which concludes with an appendix on the microscopical features of the ores by Dr. Dunn, of the Geological Survey of India.

Canadian Brass and Copper Industry

Remarkable War-time Expansion

THE review of the brass, bronze and copper products industry in Canada, just received in this country from the Dominion Bureau of Statistics, relates to 1941, the latest year for which full figures are available.

This analysis by the *Dominion Statistician*, from which this account has been abstracted with acknowledgments, shows that these non-ferrous metal-manufacturing industries have undergone a very considerable expansion during the last four years. The report deals only with those plants in which brass and copper production is the principal activity, and does not therefore include the output of iron foundries and other establishments which turn out such non-ferrous products as a by-line, or those plants which manufacture brass and copper articles for electrical industries. It relates, therefore, to firms making ingots, bars, rods, plates, sheets, discs, cups, tubing, wire cloth, valves, cartridge cases, kettles and tanks, plumbing supplies, gas and water meters, fire extinguishers, lightning rods, metal fasteners and automobile hardware.

Altogether it is estimated that there are about 325 brass plants of all types in Canada, and 143 of these are regarded as being principally concerned in the industry, 87 being actual foundries and 56 fabricating plants.

The extent of this industry in Canada may be gauged from the fact that the total value of the products of these 143 plants, which are located largely in Ontario (89) and Quebec (36) was over 100 million dollars in the year under review. This figure is more than three times the corresponding total for 1939, and twice that of 1940.

Since the number of plants has increased only by about 10% over the pre-war figure, it is evident that this very notable growth has been to a considerable extent made possible by the expansion of the existing plants. The following table summarises the present position of the industry as compared with its status in 1939:—

Number of plants—	1939.	1941.
Foundries working laps, valves, etc.	14	14
Other brass and bronze foundries	63	73
Fabricating plants	52	56
Capital invested	\$25,105,578	\$57,092,897
Employees	5,217	13,190
Salaries and wages	\$6,706,040	\$19,560,353
Fuel and electricity	\$617,235	\$1,715,107
Materials	\$18,961,128	\$59,409,859
Value of products	\$32,111,912	\$100,588,991

The analysis of the personnel aspects of this industry is also very illuminating, and some of the principal points which emerge are the increased use of female labour (1,051 against 423 in the previous year), and the fact that nearly a third of the total number of employees are working in the 56-64 hours per week category.

* "The Geology of the Nsuta Manganese Ore Deposits." By H. Service, M.Sc. Published by Gold Coast Geol. Surv. Mem. No. 5, 1943, 13½ x 8½ in. 32 pp. + 15 plates. London. Price 6s. net.

An Investigation of the Effects of Precipitation-Treatment on Binary Magnesium-Aluminium Alloys

By F. A. Fox, M.Sc., and E. Lardner, B.Sc.

Close metallographic examinations have been made of binary magnesium-aluminium alloys containing 4, 6, 8, 10 and 12% aluminium in the solution-treated and precipitated state. The effects of time and temperature have been studied, and it has been found that four general forms of precipitate are produced. Mechanical tests on precipitation-treated Elektron AZ 91 have been made, and the results show that the best properties are obtained when the precipitate is fine. The original paper is published in the JOURNAL OF THE INSTITUTE OF METALS, an abridgment of which is given here.*

THE magnesium-aluminium binary diagram has been studied, and the solid-solubility curve investigated, by many workers. The existence of a curving solubility line of the usual kind associated with a precipitation-hardening system is firmly established. This solubility change has, for many years, been utilised for the improvement by heat-treatment of the commercial magnesium alloys, which closely resemble binary magnesium-aluminium alloys in constitution and behaviour. The pseudo-eutectoid form of the precipitated beta-phase has been noted, and also that of a grain boundary precipitate, but there has been no systematic study of the metallography of the forms of the precipitate, which are numerous, and no attempt to ascertain their interrelation. The work described in the present paper had the double purpose of investigating the metallography of the precipitate, and of ascertaining whether any one particular form was to be desired, or was to be avoided from the point of view of its influence on the mechanical properties of a typical commercial alloy.

Metallurgical Examination

The specimens used in the experimental investigation were binary alloys of magnesium with 4, 6, 8, 10 and 12% aluminium, prepared from commercially pure magnesium (purity 99.8%) and aluminium (purity > 99.8%). The actual analyses are given in Table I.

TABLE I.

Non-metal Al, %	Al, %	Zn, %	Si, %	Cu, %	Mn, %	Fe, %	Sb, %	Pb, %	Ca, %
4	4.2	<0.1	0.03	0.07	0.02	<0.04	<0.03	<0.05	<0.005
6	6.3	<0.1	0.02	0.02	0.04	<0.04	<0.03	<0.05	<0.005
8	8.2	<0.1	0.03	0.02	0.03	<0.04	<0.03	<0.05	<0.005
10	10.1	<0.1	0.02	0.02	0.02	<0.04	<0.03	<0.05	<0.005
12	12.0	<0.1	0.04	0.03	0.06	<0.04	<0.03	<0.05	<0.005

Specimens for metallographic examination were cut from sand-cast D.T.D.-type test-bars, and were 1 in. in diameter and $\frac{3}{8}$ in. thick. Two specimens of each alloy were used for each precipitation temperature, one specimen being air-cooled and the other water-quenched from the solution-treatment temperature. The solution

treatment given was usually the standard one of 8 hours at 385° C. + 16 hours at 420° C., but in some cases (which are indicated), with alloys containing above 8% aluminium, solution treatments of 112 hours at 420° C. were given. Solution treatments were carried out in an atmosphere generated from sulphur, using an atmosphere circulating furnace controlled to $\pm 2^\circ$ C.

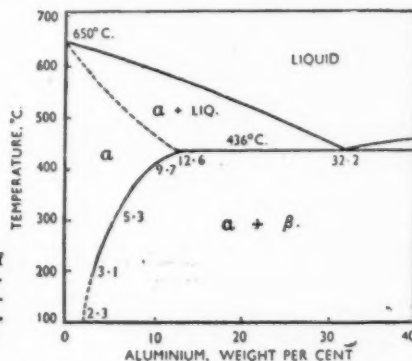


Fig. 1.—Constitution of magnesium-rich magnesium-aluminum alloys.

The procedure adopted was to subject a batch of solution-treated specimens of different aluminium contents to precipitation treatment for a given time, to examine them microscopically, and then to carry out further treatments on the same specimens. Precipitation treatments which ranged over temperatures from 130° to 250° C. were carried out in a thermostatically controlled electrically heated oven, giving an accuracy of rather better than $\pm 5^\circ$ C. Usually specimens were subjected to precipitation treatment for 6-hour periods (and multiples of this time), but in the case of the alloys with 8 and 12% aluminium the precipitation was studied in greater detail in the early stages, and treatments of 5, 10, 15 and 30 mins. were carried out in an oil-bath; in a few cases these were given for a longer time also. Specimens were allowed to cool naturally in air after precipitation treatment. With few exceptions the etching reagent employed was 2% nitric acid in alcohol.

Cast Structure.—According to the magnesium-aluminium equilibrium diagram (Fig. 1), at the eutectic

temperature of 436° C. a solid solution containing approximately 12.6% aluminium is formed; this solid solubility falls rapidly with decreasing temperature to about 2.6% at 150° C. In the sand- or die-cast alloys of magnesium and aluminium, small particles of beta-phase were produced during solidification when the aluminium content was approximately 2%. Above this the amount of beta phase was roughly proportional to the amount of aluminium in the alloy. This, of course, was only true when the alloys were cast under similar conditions; changes in rate of solidification caused differences in the amount of beta phase in one particular alloy.

The "as cast" structures often showed beta phases (usually in lamellar form) which had been precipitated from solid solution during cooling after solidification had been completed. The amount of beta phase produced in this way was usually small. It was increased by slow cooling, and occurred to a greater extent with increasing aluminium content.

Solution-Treated Structures.—Because of the increased solubility for aluminium at higher temperatures the magnesium-aluminium alloys can be improved by a solution heat-treatment, which produces a solid solution that is retained on cooling, even if only reasonably rapidly, to room temperature. It was found that the solid solution did not decompose readily, and air cooling was generally satisfactory for practical purposes. With alloys containing up to 8% aluminium the solution treatment of 8 hours at 385° C. and 16 hours at 420° C. was sufficient for complete solution of the beta-phase, but in the 10 and 12% aluminium alloys considerable amounts of undissolved beta-phase still existed. Specimens containing 10% aluminium were completely homogeneous after 112 hours at 420° C., though after the same treatment the 12% aluminium alloys still showed some beta-phase; this applied to both sand- and die-cast alloys.

Precipitated Structures.—The authors describe and illustrate the precipitated structures produced. The solution-treated alloys containing 4 and 6% aluminium showed little change in structure or hardness on precipitation treatment. The changes which took place were confined to areas, no doubt aluminium-rich, originally occupied by beta-phase in the as-cast structures, and to alterations in the etching characteristics. Thus, after solution treatment, it was not possible to develop any structure by etching with the usual nital reagent, but a fairly satisfactory grain-boundary etch could be obtained using a 5% aqueous solution of oxalic acid.

When solution-treated alloys with 8% aluminium are subjected to precipitation treatment, structural changes occur which vary considerably with the temperature at which the treatments are carried out. The changes at the early stages of precipitation were found to be masked to a considerable extent by the slight amount of precipitation which had taken place during air cooling, and in consequence these changes were observed on specimens which had been water-quenched after a long-time solution treatment. Subsequent observations showed that apart from the early stages the long-time solution treatment did not affect the precipitation. These quenched specimens, like the 4 and 6% aluminium alloys, could not be etched with nital, although a satisfactory etch was given by the same reagent when used on the air-cooled material.

The solution-treated alloys containing 10 and 12% aluminium behaved almost identically on precipitation treatment. The main difference between these alloys and that with 8% aluminium was that at the same temperatures the structures produced in the former were much finer. For this reason uniform osmondite, a minor structure in the 8% aluminium alloys, now became the predominating microstructural feature up to temperatures of about 250° C.

Forms of Precipitate

From their observations the authors distinguish four main forms of beta-precipitation. The first form comprises troosite, sorbite and pearlite; the second, crystallographic, granular and uniform osmondite; the third, spheroidic; and the fourth, mosaic outline precipitation.



Fig. 2.—8% aluminium alloy, solution-treated and quenched, precipitated 80 hours at 130° C. x 600.

Fig. 3.—6% aluminium alloy, solution-treated and quenched, precipitated 6 hours at 250° C. x 1,200.

Troosite, Sorbite and Pearlite.—These names have been used to describe precipitated structures which have the common feature that they grow from the grain boundaries of the supersaturated alpha-solid solution in the form of continuous waves. Whilst the three terms denote three types of structure, they are merely typical varieties of one main form, and it is possible to obtain all intermediate structures between two extremes reproduced in Figs. 2 and 3.

The extent of these grain-boundary precipitate areas depends upon the aluminium content of the supersaturated solid solution, and upon the temperature at which the specimen is treated. For any given aluminium content the largest grain-boundary precipitation areas are, with sufficient time, say 40–50 hours, produced at the lower temperatures. This means that in any one alloy the troosite areas for a given temperature would be larger than the sorbite areas for a higher precipitation temperature, which again would be larger than the pearlite areas for a yet higher temperature. When precipitation occurs at temperatures above 270° C. no grain-boundary pearlite is found in alloys with less than 10% aluminium, and granular osmondite is quickly produced. With alloys of more than 10% aluminium some pearlite is produced with the granular osmondite.

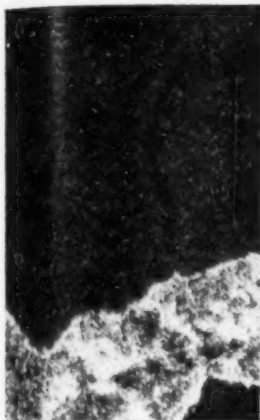


Fig. 4.—10% aluminium alloy, solution-treated and quenched, precipitated 24 hours at 150° C. x 600.

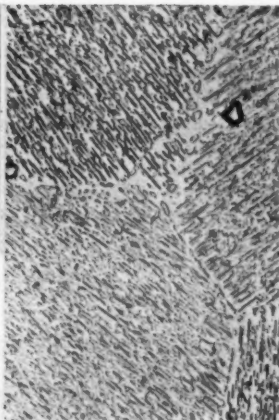


Fig. 5.—8% aluminium alloy, solution-treated and quenched, precipitated 6 hours at 200° C. x 600.

Pearlite areas seen in alloys with less than 10% aluminium treated at temperatures in this region are almost certainly produced during the heating period. The upper limit of the temperature range in which pearlite precipitation occurs increases with increasing aluminium content.

For a fixed precipitation temperature the largest grain-boundary-precipitation areas appear to be produced in an alloy containing about 9% aluminium. Above and below this aluminium content the areas decrease.

Osmondite Precipitation.—Osmondite, in its three forms, crystallographic, uniform and granular, comprises the precipitates which are produced in areas not affected by the growth of the grain-boundary precipitate.

In an alloy containing up to 12% aluminium that has undergone precipitation treatment at any temperature, the first appearance of osmondite is always in the crystallographic form. This precipitate is not produced immediately on reheating, but requires an "incubation" period which varies with the temperature and the degree of supersaturation of the alloy. In a 12% alloy treated at 130° C. the period is about 80 hours, but with increasing temperature it rapidly shortens, being about 1 hour

at 150° C., less than half an hour at 250° C., and about 6 mins. at 300° C.; at 350° C. it was found to be too short to determine. An 8% aluminium alloy treated at 150° C. shows the initial formation of crystallographic osmondite after about 11 hours. During this incubation period troosite, sorbite or pearlite areas are being formed in the grain-boundary regions, and it has not been possible at any temperature to produce a crystallographic osmondite structure alone. Uniform osmondite is produced from crystallographic osmondite by continued treatment at temperatures below about 200° C. (Fig. 4). It seems that the precipitate remains in the form of extremely small particles, presumably because of the low atomic mobility at these lower temperatures.

On continued heating of crystallographic osmondite above about 200° C. the orientation effect becomes more marked and the particles coalesce, forming parallel layers (Fig. 5), which, from the observed variation in thickness from grain to grain, apparently consists of a series of parallel plates.

Another form of precipitate, which is probably crystallographic in type, can be seen in Fig. 5, where it appears as small bars crossing the basal plane precipitation at approximately right angles.

The mosaic type of precipitation, reproduced in Figs. 6 and 7, appears early in the course of precipitation treatment, and occurs most markedly in specimens of large grain size, especially in specimens in which a great amount of grain growth has taken place during the solution treatment. There is a tendency for a large proportion of this precipitate to be parallel to directions in which osmondite is eventually formed. Individual particles in the streaks of precipitate tend to be elongated in form in the same direction.

It appears that mosaic precipitation may occur as a result of beta-particle precipitation on the sites of original grain boundaries. It is known that, in general, precipitation rapidly occurs on the boundaries of twins, where the latter structure is faulted, and at true grain boundaries, where there are lattice disturbances. It is also probable that when grain growth takes place some lattice imperfection still remains on the sites of the original grain boundaries, and this serves as the basis for early precipitation in the case of structures such as that of Fig. 7.

Rate of Precipitation

It is not easy to estimate rates of precipitation and the time required for complete precipitation by observation of microstructures and by hardness measurements. However, it is evident that the rate of precipitation increases very rapidly with increasing temperature, and also with increasing aluminium content. As far as composition is concerned the controlling factor will be the degree of supersaturation existing at the particular precipitation temperature, which for any given alloy will decrease with increasing temperature. Consequently the higher rate of precipitation caused by increasing temperature will be offset to some extent by the accompanying reduction in degree of supersaturation.

Rate of precipitation is also influenced by grain size. As indicated above, the nucleating influence of the grain boundaries gives rise to a higher ratio of grain-boundary precipitation to osmondite precipitation as the grain size falls. This results in the precipitation taking place more rapidly with decreasing grain size. It has been shown that long incubation periods are required before precipitation will occur in single-crystal specimens

Fig. 6.—8% aluminium alloy, solution-treated and quenched, precipitated 6 hours at 150° C. x 600.



Fig. 7.—8% aluminium alloy, solution-treated and quenched, precipitated 16 hours at 240° C. x 600.



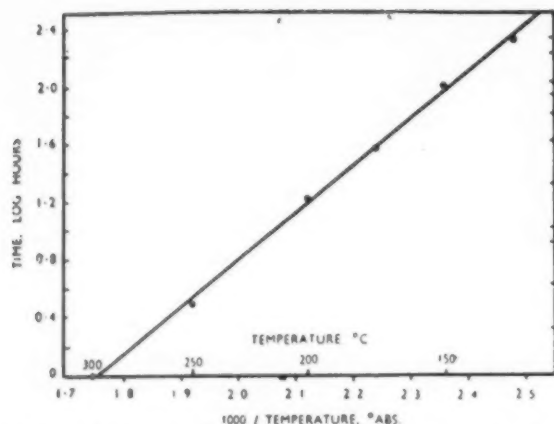


Fig. 8.—Time to produce maximum hardness v. temperature for Elektron AZ 91.

of magnesium-aluminium alloys, and the same tendency with increasing grain size has been noted during the course of the present work.

In dealing with the mechanical properties it was necessary to determine, approximately, the time required to produce the maximum values at various temperatures for Elektron AZ 91 (closely resembling a binary aluminium-magnesium alloy containing 10% aluminium). This was done on sand-cast test-bar samples (grain size approximately 0.1 mm.), and the results are given in Fig. 8.

The log of time in hours has been plotted against the reciprocal of the absolute temperature. This graph, on extrapolation, suggests that the period required for maximum hardening at 100° C. would be 40 days if the necessary changes could occur at all, and at room temperature (15° C.) about 40 years. These indications are of some interest in view of various comments which have been made regarding the room-temperature ageing of magnesium alloys.

It is to be expected that the rate of precipitation will be increased by cold-working the solution-treated material before precipitation treatment. During the course of the present metallographic work difficulty was experienced owing to precipitation occurring on twin markings produced by straining the solution-treated specimens while preparing the surface for micro-examination. Precipitation occurs in these regions at a similar rate to that in the grain boundaries.

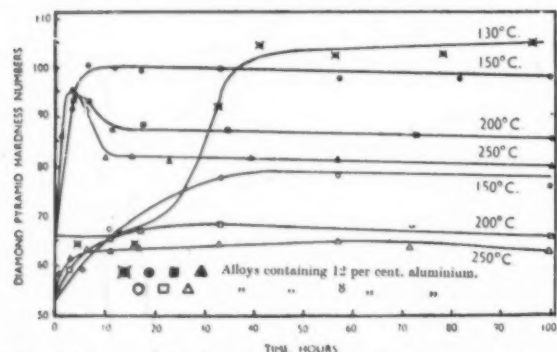


Fig. 10.—Precipitation hardening of two magnesium-aluminium alloys at 130°, 150°, 200° and 250° C.

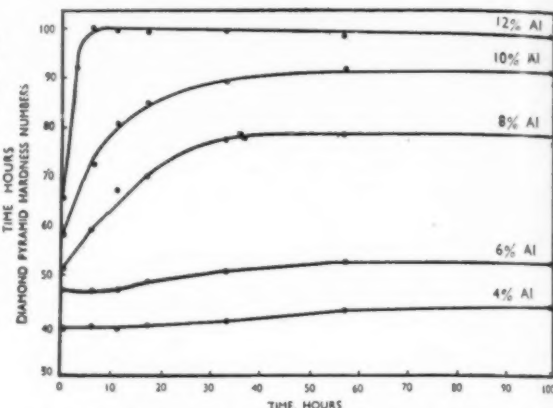


Fig. 9.—Precipitation hardening of magnesium-aluminium alloys at 150° C.

Mechanical Properties

The effect of all the precipitation treatments upon the hardness figures of the alloys used for this investigation was measured. Each hardness figure given represents the mean of at least six determinations; occasional low-hardness figures were rejected. The original intention of making hardness measurements upon the micro-specimens was found to be undesirable, owing to some local non-uniformity in hardness of the individual specimens. It was, however, found possible to obtain reasonably consistent results on specimens cut from the base of blocks cast in a water-cooled mould. The hardness figures shown in Figs. 9 and 10 were obtained by using such specimens. The grain size of these was 0.06 to 0.09 mm., whereas for the cast specimens described earlier it was about 0.1 mm. Fig. 11 shows graphically the relation between the hardness changes and structural changes in 8 and 12% aluminium alloys cast in the water-cooled mould. It will be seen that the structural changes exert no sharp influence on the hardness numbers. Figs. 9 and 11 show that:—

1. The rate of hardening increases with increased aluminium content.
2. The degree of hardening produced with less than 8% aluminium is very small.

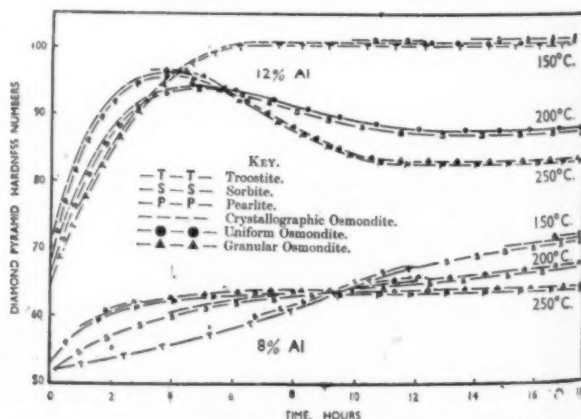


Fig. 11.—Structural and hardness changes on precipitation treatment of two solution-treated magnesium-aluminium alloys.

Fig. 10 shows the influence of temperature upon the hardening of the alloys containing 8 and 12% aluminium. This figure shows that:—

1. Maximum hardness is obtained by precipitation at the lower temperatures, but longer times are required.

2. The initial rate of hardening is higher at the higher temperatures.

3. For each temperature, after a peak hardness value has been reached, further treatment results in softening. This is most marked for the 12% aluminium alloy and the higher temperatures.

4. The 12% aluminium alloy, when heated at 130° C., shows no hardening in the initial stages of heat-treatment.

These results show that it is inadvisable to use the high-aluminium alloys for applications where they will be run at temperatures much above 150° C.

With regard to tensile properties, the structures produced on the precipitation treatment of solution-treated magnesium-aluminium alloys are of practical importance in the case of the Elektron AZ 91 (aluminium 9.5–10%, zinc 0.4%, manganese 0.3%), which is frequently used in the precipitation-treated state. Observations of structural changes produced by varying time and temperature of precipitation treatment were made for this alloy; it was found that the structural and hardness variations were the same as for the 10% aluminium alloy, the small amount of zinc making no observable difference. On the basis of the hardness results the precipitation treatments which would produce the best mechanical properties in solution-treated Elektron AZ 91 were estimated. After several pilot treatments had been carried out, the following treatments were chosen:—

(a)	Solution treatment	+ 200 hours at 130° C.
(b)	"	" + 96 " 150° C.
(c)	"	" + 36 " 175° C.
(d)	"	" + 16 " 200° C.
(e)	"	" + 6 " 230° C.
(f)	"	" + 2 " 300° C.
(g)	"	" + 72 " 250° C. (spheroidised structure).

Heat treatments (a), (b), (c), (d) and (e) were applied to one batch of D.T.D.-type Elektron AZ 91 test-bars cast from one melt (C. 389), whilst heat treatments (b), (c), (d), (e), (f) and (g) were applied to D.T.D. type test-bars cast from another melt (C. 350). Each batch of bars was from the same melt, and six bars were taken from each precipitation treatment.

Test-bars taken from melt C. 350 were given the standard solution treatment of 8 hours at 385° C. and 16 hours at 420° C., whilst the bars from melt C. 389 were given 8 hours at 385° C. + 64 hours at 415° C. There was little difference in grain size, but the degree of solution in the second batch of brass was much higher. The analysis of the two melts of Elektron AZ 91 used is given in Table II. The average results from six bars from each treatment are shown in Fig. 12.

TABLE II.

Melt No.	Al, %	Zn, %	Si, %	Cu, %	Mn, %	Fe, %	Sb, %	Pb, %	Ca, %
C. 389	9.6	0.45	0.03	0.03	0.21	<0.04	<0.03	<0.05	<0.005
C. 350	10.1	0.46	0.04	0.04	0.21	<0.04	<0.03	<0.05	<0.005

The results obtained are intended to represent the maximum properties obtainable at the particular temperature chosen. However, maximum proof stress does not of necessity correspond exactly with maximum

hardness, and the treatments chosen may not exactly coincide with those required to produce the best properties. It has been shown by past experience that the effect of over-precipitation on the tensile properties is not great at temperatures below 200° C., and the present work has shown that the difference in properties between precipitation for 6 hours and for 72 hours at 250° C. is very slight. As the times chosen are believed to be generous, it seems that the curves shown in Fig. 12 can be accepted as reasonable illustrations of the effect of temperature on tensile properties. The following information is given by these curves:—

1. The ultimate tensile stress is greatly increased by the much-prolonged solution treatment, the improvement for all precipitation treatments being of the order of 3 tons/sq. in.

2. Prolonged solution treatment causes a slight decrease in the 0.1% proof stress obtained in the precipitated condition.

3. Prolonged solution treatment causes an increase in the percentage elongation in the precipitated condition.

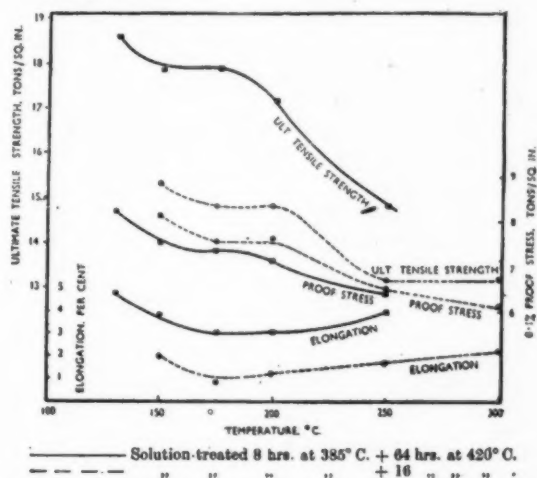


Fig. 12.—Tensile properties of precipitation-treated Elektron AZ 91. Time of treatment chosen to give maximum hardness at each temperature. Each point is the mean of six results.

4. Increasing precipitation temperature causes reduction in ultimate and proof stresses, with but little corresponding improvement in percentage elongation, which actually seems to pass through a minimum at about 175° C. The drop in properties is not great between 150° and 200° C., but above 200° C. the ultimate tensile stress falls off rapidly. This coincides with the formation of granular osmondite.

The results obtained show clearly that the best mechanical properties are associated with fine well-dispersed beta-phase. Thus, the specimens treated at 130° C. showed a structure of troosite + very fine osmondite. Treating at 150° C. produced a structure of sorbite and rather coarser osmondite, with a corresponding decrease in proof and ultimate stresses. The structure produced at 175° C. is very similar.

Precipitation treatment at 200° C. produced pearlite and coarse osmondite, whilst the bars treated at 250° C. showed coarse pearlite and granular osmondite, a com-

bination giving comparatively poor properties. It is interesting to note that the conversion of granular osmondite to a spheroidised structure causes practically no change in tensile properties, although it might have been expected to cause some decrease in proof and ultimate stresses with an increase in elongation.

The high properties obtained by a combination of prolonged solution treatment and long-time low-temperature precipitation treatment are in themselves very interesting, apart from the prohibitive length of the heat-treatment times required. The 5% elongation is of

particular interest, in relation to the more normal lower elongation figures which constitute the drawback of using material in the precipitated condition. This improvement in elongation is probably associated as much with the long-time solution treatment as with the low-temperature precipitation treatment.

It will be noted that the mechanical properties change smoothly, falling with the gradually increasing coarseness of the precipitate; no sharp change occurs on the appearance of any one type of precipitate, nor is any one type in itself (apart from its fineness) especially valuable.

Improved Temperature Regulators

TWO instruments of improved design for temperature regulation have recently been developed by Cambridge Instrument Co., Ltd. One of these instruments is a compact self-contained thermometer regulator developed for automatically controlling the temperature in a wide variety of applications at any point between the limits of -20°C . and $+600^{\circ}\text{C}$.

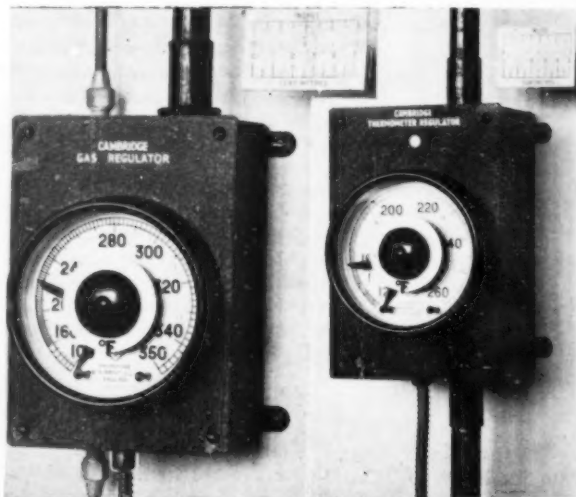
This regulator consists of a 4-in. dial thermometer provided with an adjustable contact by means of which a self-contained tilting mercury switch controls the electric heating supply. A feature of the instrument is that while the thermometer contacts carry only little current, the self-contained switch operated by them is capable of carrying a maximum current of 15 amp. a.c. or d.c. at any voltage up to 250.

The dial thermometer, together with the mercury switch, is contained in a rectangular metal case provided with terminals for connecting to the load and supply, no separate accessory apparatus, such as external contactors or relays, being required. The sensitive bulb of the instrument, which is inserted in the tank, oven, or platen, etc., and thus exposed to the temperature being controlled, is connected by a length of flexible tubing to the pointer mechanism. The bulb connection is made in various forms, to suit different applications, and the flexible tubing may be any length up to 100 ft., thus enabling the dial to be placed in the most conveniently accessible position.

The adjustable contact on the thermometer dial is spring-mounted on a movable red index, which is set by a detachable key to the temperature at which control is desired. When the actual temperature is below this point the thermometer contacts are closed and the mercury switch is held in the "ON" position. When the pointer reaches the control temperature the thermometer contacts open and the mercury tube tilts to the "OFF" position, thus switching off the main supply. The position of the mercury switch is indicated by a coloured "flag" through a small window above the dial; this flag shows red when the current is "ON," and white when switched "OFF." Since the main contacts of the switch require an electric current to close them, the arrangement has the advantage that in the event of any fault developing in the internal wiring the main contacts will immediately open circuit and prevent damage to the process through overheating.

The second instrument is designed to indicate and control the temperature of gas-heated apparatus, and is available in various ranges covering -20°C . to $+600^{\circ}\text{C}$. This instrument comprises a 4-in. dial thermometer fitted with adjustable contacts controlling a small solenoid valve which is operated off the electric

mains supply. The solenoid valve, which is accommodated with the thermometer dial in a rectangular metal case, in turn operates a relay gas valve inserted in the main gas supply pipe-line. These relay valves are available in nine sizes, suitable for pipes covering a



A compact regulator for gas-heated processes for use with standard relay gas valve.

An improved self-contained thermometer regulator.

range from $\frac{1}{2}$ in. to 3 in., and can be employed for pressures varying from a few inches water gauge to 4 lb. per sq. in. The adjustable contact on the thermometer dial is spring-mounted on a red movable index which is set by a detachable key to the temperature at which control is desired. When the temperature-indicating pointer reaches this index the solenoid valve is operated and the gas supply is shut down. The flexible tubing connecting the sensitive bulb and the thermometer dial is usually 6 ft. long, but may be any desired length, enabling the dial to be erected in the most conveniently accessible position, away from fumes or heat. When installed in positions subject to corrosive action the tubing and bulb can be made of stainless steel, or otherwise suitably protected.

An internal by-pass is incorporated in the relay valve, which prevents the burner from being extinguished when the control is operated to close the valve. The connections between the regulator and the relay valves are made by copper tubing $\frac{3}{16}$ in. inside diameter by $\frac{1}{4}$ in. outside diameter, this tubing can be supplied to order, if desired.

Radiography of Metals and Alloys

Review of an American Symposium

Modern industrial radiology embraces two main fields, the first the examination of the internal soundness of metals and alloys by radiography with both X-rays and gamma-rays, and the second the examination of the crystal structure of metals by X-ray diffraction. Radiography is a non-destructive method of testing, and is one in which many developments have taken place during recent years. In 1936 a symposium on Radiography and X-ray Diffraction Methods was held by the American Society for Testing Materials, and a number of papers were presented dealing with the best practice in use at that time. Last year a second symposium on Radiography¹ was held by the same Society, at which a series of papers dealing with modern practice were presented. These papers cover a wide field and deal with the application of X-ray inspection to production problems, the radiography of welds, castings, boiler and pressure pipe lines, and to developments in modern radiographic equipment.

Recent Industrial Applications

IN discussing some applications of X-ray inspection to production D. McCutcheon considers that the problems arising from such methods may be ascribed to two specified causes. One is the high-volume production of cast light alloys and other metals required for aircraft construction, and the second the extension of high-strength steel castings into new fields formerly dominated by forgings. Special applications and Government specification probably also account for a considerable extension of X-ray inspection in some fields. Application of X-ray has also multiplied many times during the last two years, and is considered likely to increase as the special demands of the armament programme increases.

Some practical applications to large volume production inspection are discussed for both low-voltage and high-voltage X-ray practice, both for light metals and alloys and for steel and other alloys. The equipment and technique suitable for the X-ray examination of aluminium and magnesium alloys requires the careful consideration of a number of factors. Low X-ray absorption must be compensated for by the use of low-voltage or long-wave-length X-rays, and the size and distribution of defects encountered require that a reasonable degree of image sharpness be obtained. The procedure best followed in radiographing light alloy castings is to use the lowest voltage on the X-ray tube, consistent with reasonable exposure time, and fine-grained high-contrast X-ray films, together with correct developing technique. It is also pointed out that the successful examination of complex-shaped castings of aluminium and magnesium alloys also require considerable care in selecting the proper angles and position of the casting with respect to the direction of the X-ray beam. The X-ray examination of steel and copper alloys requires high X-ray intensity, and one of the main problems encountered with the use of X-ray beams generated by voltages between 100 kv. and 700 kv. is scatter radiation. For relatively thin sections and moderate voltages, filters interposed between the object and the film are sufficient to markedly reduce the scattered X-ray intensity emitted by all objects in

the path of the primary rays, Table I. For irregular-shaped castings a double-filter technique has been found advantageous. The thickness of metal which may be examined by X-rays between 150 kv. and 400 kv. may be extended by the use of calcium-tungstate screens, but the necessity for the use of such screens is eliminated by the use of the million-volt X-ray unit.

TABLE I.—RECOMMENDED X-RAY FILTRATION (MCCUTCHEON).

Kilovoltage.	Application.	Section Thickness, In.	Filter.
15—60	Magnesium alloys.	$\frac{1}{8}$ to $\frac{1}{4}$	No filter at tube or film.
30—60		$\frac{1}{8}$ to $\frac{1}{4}$	No filter at film, copper $\frac{1}{8}$ mm. thick at tube for special sections optional.
60 to 100	Aluminium alloys.	$\frac{1}{8}$ to $\frac{1}{2}$	No filter for plain sections $\frac{1}{8}$ mm at tube for complex shapes 0.002 to 0.005 in. copper foil at film, optional.
140		Above $\frac{1}{2}$	Copper or lead filter 0.005 in. thick at film surface.
125 to 250		$\frac{1}{8}$ to $\frac{1}{4}$	Lead filter at film surface 0.005 mm. thick. Lead filter at tube 0.010 in., optional.
220 to 400		1 to $2\frac{1}{2}$	Lead filter 0.005 to 0.025 in. thick at film.
1,000	Steel	$2\frac{1}{2}$ to 5	Lead filter 0.010 in. tube, optional. Lead filter 0.005 to 0.250 — at film, depending upon irregularities of section. Lead filter up to $\frac{1}{8}$ in. at tube for round and irregular sections.

After describing automatic X-ray units, and the technique used for the examination of large quantities of small aluminium and magnesium castings, and also the million-volt X-ray unit, McCutcheon states that the advent of this latter equipment brings X-ray radiography within the range of metal penetration and effective quality of radiation with radium. The use of radium is still extending, however, owing to the low cost per unit examination, and its flexibility of use with the small capsule.

Other applications of radiography dealt with are in the examination of spot-welding of thin aluminium sheet and microradiography, a radiographic process which resolves structural details in metals and other objects by X-ray absorption formerly confined to the optical microscope. X-ray technique for obtaining maximum

¹Trans. Amer. Soc. Testing Materials, 1942, vol. 42, pp. 1,023-1,182.

details and sharpness of films of spot welds requires the use of low-voltage X-ray beam (15 kv. to 30 kv.), small focal area X-ray tubes and high-contrast fine-grained films. Microradiographic technique involves the careful preparation of metal samples, low-voltage radiation, and the use of suitable films depending on the magnification, and new methods and applications for this process are outlined.

Examination of Welds

Developments in the general art of radiographing of welds which have come into use during the last six years are reviewed by R. E. Lorentz, who points out that the raising of the voltage from 400,000 v. to 1,000,000 v. has brought about marked improvements in technique by shortening the exposure time from hours to minutes, and by raising by many inches the maximum weld thickness which can be radiographed. Such developments have not only allowed the wider use of lead intensifying screens, but have also brought about techniques such as double-film viewing, where an exposure is made with two films in the same cassette, and which presents many advantages such as eliminating retakes due to film defects, and offering an advantage in radiographing differential thicknesses. New fine-grained high-contrast films have also come into use, and these improve greatly the detail obtained in weld radiographs, particularly in the field of gamma-radiography. Changes in the A.S.M.E. Boiler Code for fusion welds requirements are also discussed, particularly the penetrometer or sensitivity gauge of uniform thickness, now more or less standard, which has replaced the step type of penetrometer. The type of weld defects porosity and slag inclusions which should be allowed and which should be removed are also considered in view of the wider experience gained during recent years.

The application of high-voltage X-rays in the boiler-shop for the examination of fusion welds of main longitudinal and circumferential seams of heavy-walled pressure vessels with plate thicknesses up to 5½ in. are dealt with by O. R. Carpenter, who shows that until the million-volt X-ray equipment becomes available for industrial purposes the highest voltage equipment in general industrial use was 400 kv., and with it approximately 15 days were required for the complete radiographic examination of a boiler-drum, although some time could be saved if gamma-rays were substituted for X-rays for the circumferential seams, the difference being from two days upwards, depending upon the size of radium capsule available. Application of higher-voltage X-ray equipment to industrial radiography introduced the problem of blocking excessive secondary radiation from the film, and this requirement was met by the development of the Bucky grid and similar devices, and although a considerable increase in exposure time resulted from the use of these grids, X-ray films of good detail were obtained with 300 kv. and 400 kv. equipment.

With the advent of the million-volt X-ray equipment with the emission of extremely short-wave-length X-rays a radical change was necessitated in X-ray technique. Special fine-grained no-screen films (direct-exposure films) were developed for this purpose, and were a significant advancement in the solution of radiographic problems. The use of lead-foil screens in conjunction with these films was considered to warrant the following

change in X-ray boiler-shop technique: (1) The Bucky grid need no longer be considered an essential part of X-ray technique; (2) the calcium-tungstate screen exposure of X-ray films could be discarded; (3) proper voltages and film combinations were essential for good radiographs of fixed ranges of plate thicknesses; (4) the finer-grained film made possible the successful use of shorter wave-lengths, even shorter than those of the million-volt apparatus, and gamma-rays gave excellent results; (6) X-ray techniques might be standardized, and, if followed, satisfactory results were assured.

Radiography of Castings

A method for the quantitative rating of radiographs showing porosity in magnesium-base alloys was described by R. S. Busk. By this method the intensity of the viewing light is varied with a voltage Variac, and the voltage noted at which porosity is just visible. A scale is established for the rating of radiographs, known as the "contrast index," denoted by the symbol $1/v$, and having values from about 200 at just detectable porosity to values above 400, and this scale is used to study the effect of micro-porosity on the tensile properties of Dowmetal H alloy in its as-cast and solution heat-treated conditions; the tensile properties of Dowmetal C alloy in its as-cast solution-treated and heat-treated and aged conditions, and the fatigue strength of the C alloy in its heat-treated condition. As the result of this investigation it was found that the tensile strength of magnesium alloys decreased as the amount of porosity increased, and that the yield strength of magnesium-alloy castings was very little affected by porosity. A $1/v$ rating of 300, representing an amount of porosity that is heavier than that usually obtained in the foundry, represented a tensile strength of the solution-heat-treated H alloy within the specified minimum of bars cut from castings. C alloy was found to be less porous than H alloy under similar foundry conditions, but with a given amount of porosity. H alloy suffered a smaller loss of strength than C alloy. All the alloys were more sensitive to porosity in the solution-heat-treated condition than in the as-cast condition. The fatigue strength of solution-heat-treated C alloy was found to decrease with increased porosity to the same degree as did the tensile strength for a given amount of porosity.

The radiography of magnesium-alloy castings for aircraft purposes is discussed in a paper by L. W. Ball, who deals with some calibration data and scatter measurements for such castings. Penetration data is best obtained either by a calibration-graph showing the kilovoltage required to penetrate a given thickness of magnesium with a selected exposure factor, or by a curve showing the exposure factor required to penetrate a given thickness of magnesium with a selected kilovoltage. For the radiography of magnesium-alloy sheet of constant thickness, where it is important to determine how sensitivity varies with kilovoltage, measurements are made by film density differences, and for the radiography of castings where a considerable range of thicknesses occur calibration curves are used which show the range of metal thickness over which sensitivities of 1, 2, 5 and 10% are obtained. As no type of calibration is suitable for all the various shapes of castings, magnesium aircraft castings are divided into three groups on the manner and degree to which secondary radiation

spoils the primary sensitivity. An appropriate calibration must be chosen for each group such that, when a casting is placed in the proper group, scatter does not reduce the claimed penetrometer sensitivity by more than twice. For "well-blocked" castings suitable calibration is a matter of determining what exposure factor, screens and film will give the maximum sensitivity within the practical limits of exposure time, and for "baldy-blocked" castings it is essential to use a relatively low-exposure factor and a front filter screen.

An experimental method is also described for the measurement of scatter based on the principle that the primary X-ray intensity is independent of the field size, but that scatter intensity is not. Curves obtained show that the ratio of the scatter to the transmitted intensity increases approximately linearly with thickness. This ratio is important in the radiography of uniform sections, but for "baldy-blocked" castings the image of one section can receive scatter from other sections of different thicknesses, so that it is essential to know how the intensity of this scatter depends on the thickness of the section producing it. It is also stated that weight for weight magnesium and aluminium actually produce very nearly the same amount of scatter.

Examination of High-Pressure Piping

The gamma-ray radiography of welded high-pressure power piping is dealt with by R. W. Emerson, who considers the apparatus necessary, and the procedure used in the gamma-ray radiography of both circumferential and longitudinal seams in welded piping. Also discussed is the size of radiant source, distance from the source to film, metal thickness, and type of film on the resultant radiographic sensitivity. Gamma-ray radiography is considered preferable to X-ray radiography in examining circumferential pipe welds due to its simplicity and greater adaptability, particularly where field radiography is required. Several procedures used in setting up for radiography of both types of seams in piping are considered, and an investigation is made of absolute radiographic sensitivity as effected by source to film distance, metal thickness and source intensity.

The determination of these factors was made by an accurately drilled test-block, having three series of holes of $\frac{1}{16}$ in., $\frac{1}{8}$ in. and $\frac{3}{16}$ in. diameter and depths of 0.010 in. to 0.100 in., being radiographed using many combinations of source to film distance and metal thickness, and using both a 25-mg. and a 50-mg. radium source. The results obtained are given in Table II and show that the smaller radium source definitely proved superior to the larger for short source to film distances, but offered little if any advantage as the source-to-film distance increased beyond 6 in. As a result of these tests it is considered that when radiographing circumferential pipe welds in which the radium is placed centrally, the source-to-film ratio should seldom exceed 7 to 1, and might be as low as 3 to 1. When working with such low ratios the size of the radium source should never exceed 50 mg., and should preferably be 25 mg., if good radiographic detail is to be expected.

In another determination two circumferential test pipe welds were made in which many defects, common to welded piping, were purposely made. These welds were first radiographed and then sectioned in many

TABLE II.—ABSOLUTE RADIOGRAPHIC SENSITIVITY AS EFFECTED BY SOURCE-TO-FILM DISTANCE, METAL THICKNESS AND SOURCE INTENSITY (EMERSON).

Radium Capsule	Distance, In.		Ratio A — B	Depth of Last Hole Visible, In.			Remarks.
	(A) Source to Plane of Holes.	(B) Plane of Hole to Film.		$\frac{1}{16}$ in. hole.	$\frac{1}{8}$ in. hole.	$\frac{3}{16}$ in. hole.	
25	1.75	0.50	3.50	0.01	0.01	0.01	Hazy, 0.01 very faint.
	3.50	0.50	7.00	0.01	0.01	0.01	Better detail than No. 1, 0.01 very faint.
	3.50	1.00	3.50	0.02	0.02	0.02	Hazy, enlargement of image on holes.
	5.25	0.50	10.50	0.01	0.01	0.01	Sharp image, 0.01 very faint.
	5.25	1.00	5.25	0.02	0.02	0.02	Sharp image, slight enlargement.
	5.25	1.50	3.50	0.03	0.03	0.03	Very hazy, enlargement of image.
	7.00	0.50	14.00	0.01	0.01	0.01	Extremely sharp image, 0.01 faint.
	7.00	1.00	7.00	0.03	0.02	0.02	Sharp image.
	7.00	1.50	4.67	—	—	—	Under-exposed.
100	1.75	0.50	3.50	0.02	0.02	0.02	Much more hazy than No. 1.
	3.50	0.50	7.00	0.02	0.02	0.02	Hazy, but improved over previous exposure.
	3.50	1.00	3.50	0.03	0.03	0.03	Very indistinct and hazy.
	5.25	0.50	10.50	0.02	0.01	0.01	Sharp image, good detail.
	5.25	1.00	5.25	0.03	0.02	0.02	Hazy image, 0.02 faint.
	5.25	1.50	3.50	0.03	0.03	0.03	Very hazy and enlarged image poor, 0.03 faint.
	7.00	0.50	14.00	0.01	0.01	0.01	Sharp image, good, 0.01 faint.
	7.00	1.00	7.00	0.03	0.02	0.02	Hazy but fair detail, 0.02 faint.
	7.00	1.50	4.67	0.03	0.03	0.03	Fair detail, 0.03 faint.

places, polished and etched and used to check the actual defect with the radiograph. Two types of penetrometer were placed on both the source side and film side of these pipe welds, and it was found that there was little if any difference with the penetrometers in the two positions, at least for wall thickness up to 1.5 in. It appeared, however, that penetrometer detail was slightly better when the penetrometers were placed on the film side, although this did not give an absolutely true picture of the detail of a defect comparable to the size of the penetrometer holes which is close to the root of the weld.

The A.S.M.E. Code for the fusion of welding of pressure piping requires that in radiographic testing, penetrometers must be placed on the inside of the pipe. On this account welded pipe joints are difficult to radiograph when the diameter is small. R. J. Schier and G. E. Doan have investigated the possibility of using penetrometers next to the film instead of next to the source in radiographing pipes, and the thickness of these penetrometers to make them equivalent to those used in the code were determined experimentally. The data obtained shows that in order to be equivalent to the standard penetrometers for specimen thicknesses of $1\frac{1}{2}$ in., $1\frac{3}{4}$ in. and 2 in., the equivalent penetrometers should have, respectively, approximate thicknesses as follows—0.0142 in. instead of 0.030 in., 0.020 in. instead of 0.035 in., and 0.025 in. instead of 0.040 in. When an equivalent penetrometer is used, the radiographic conditions must be the same as those used when the penetrometer is located in its usual position on the specimen surface nearest the source.

Developments in Radiographic Equipment

A million-volt X-ray unit of small dimensions and light weight is described by E. E. Charlton and W. F. Westendorp. Its principal features, the 12-section

X-ray tube for continuous operation at 1,000,000 v., the coreless resonance transformer, and the Freon gas (dichloro-difluoro-methane gas) insulation are dealt with in detail. This unit permits radiography from ten to several hundred times faster than with older equipment through thick masses of steel ranging from $\frac{1}{4}$ in. to 8 in., producing a radiographic definition of 1 to 2%. The intensity and penetrating power of the beam permits greater source to film distances to be used, and still keeps the time of exposure down to seconds and minutes and thereby markedly reduces the distortion of the shadows recorded on the film, and also increases the area of the material which can be radiographed with one-exposure technique. Stereoscopic radiographs of metal sections of widely varying thicknesses may also be obtained with good definition, this being made possible and practical as a result of the large amount of penetrating X-radiation which is generated by this unit.

The results of an investigation of the apparatus used in radium radiography are given by L. W. Ball and D. R. Draper to provide guidance to radiographers in the selection and setting-up of their apparatus by measuring the extent to which variation in each feature of the apparatus affects radiographic sensitivity. The features considered are: (1) The source, consisting of the active substance and the capsule in which it is sealed; (2) the surroundings, together with the protective housing used to limit the spread of radiation; (3) the cassette containing the film and screens, and (4) the photographic equipment. Experiments show that the effect of source, size and secondary effects can be neglected, that the relative legibility of narrow images can be anticipated by calculating the simple umbra limits, and that the shape of capsule should be spherical. Operating surroundings should be tested for surface scatter by means of a lead pyramid arrangement, and should be reduced to a minimum. The design of a suitable cassette depends on the thickness of the lead screens, and a compromise screen thickness of 0.004 in. has proved suitable for general use with thick castings but under conditions where scatter absorption is relatively unimportant thin screens are of general use. Direct exposure films give the greatest available speed contrast and radiographic range, but have the disadvantage of a larger grain size.

L. W. Ball also presents a paper on a study of cassette design for the radiography of aircraft castings in which a systematic determination is made of the features which are essential in such design to prevent scatter radiation from the cassette material from spoiling radiographs. Experiments carried out on screen film cassette design and on direct exposure film cassette design are considered, and a cassette intended for a direct exposure film is described which incorporates all the features necessary to reduce scatter to a minimum. It consists of a brass periphery with 0.005 in. of steel, 0.003 in. of lead, the film, 0.021 in. of lead, $\frac{1}{8}$ in. of felt, and a brass pressure plate. The relation of cassette design to radiographic practice is also considered in respect to the selection of suitable equipment and in the selection of calibration techniques for each class of work. Different cassettes, together with their screens, are compared for speed sensitivity and range by preparing radiographs of a slotted wedge penetrometer.

* An X-ray exposure meter developed for use with a 200-kv. X-ray radiographic installation in use at the United States Naval Research Laboratory is described by H. Friedman and A. L. Christenson. For given brands of film and intensifying screens, this meter may be calibrated to indicate the exposure times required for any desired film blackening and the dependence of this calibration on kilovoltage, specimen material, and filter technique is investigated. In routine radiography it is found possible to obtain satisfactory results with a single calibration curve at all voltages between 125 kv. and 200 kv. for any specimen material, and for filtered as well as unfiltered radiation. The receiving element of this exposure meter is a Geiger-Muller counter, designed to permit accurate intensity determinations in a few seconds, with the measured beam of radiation limited to a pencil of X-rays, $\frac{1}{4}$ in. in diameter. The use of a small aperture on the counter permits the scanning of portions of castings on non-uniform thickness to determine an optimum kilovoltage and suitable filler technique for radiographing. As the counter is placed close to the film position the meter indications are independent of variations in the source-to-film distance.

Precision Radiography and Film Evaluation

Precision radiography is dealt with in a paper by R. J. Schier and G. E. Doan, who discuss the general equation which has been developed for calculating radiographic distances. This equation is considered rather cumbersome for ordinary radiographic problems, and especially for those in which the A.S.M.E. Code is followed and a special application of it is developed by these authors and expressed as—

$$d = \frac{R + r}{\sin \alpha/2}$$

where d is the source to specimen distance; R the radius of the source; r the radius of the flaw, the smallest hole in a penetrometer that lies on the surface of the specimen nearest the source; and α the common vertex angle subtended by the source and the flaw. To test this equation, and to determine the angle α which should be adopted for certain registry of A.S.M.E. penetrameters lying on the shortest line between the source and the film, a series of exposures were made using gamma-rays from a spherical source having a working diameter of 3.0 mm. A vertex angle of $1\frac{1}{2}^\circ$ was obtained for specimen thickness up to 2 in.

To use and improve the recording medium of industrial radiography more effectually, it is necessary to evaluate the characteristics of such material quantitatively, and this is done by V. Danford, who has worked out a series of methods for interpreting the results of controlled X-ray exposures. Graphs are given showing basic X-ray exposure plotted from densities produced from various thicknesses with all other factors unchanged; a voltage thickness relation with density at 1.0 and exposure times varying from 15 to 120 secs., a voltage thickness relation with constant exposure, and densities ranging from 0.5 to 4.0; a basic X-ray exposure with contrast values, a density exposure time relation for constant thickness, a voltage-thickness relation showing contrast, and radiographic contrast comparison for two types of films with constant voltage and constant time, respectively.

Microchemical Analysis

SINCE the beginning of the war, when the Microchemical Club suspended its activities, microchemists in this country have, for the most part, been forced to depend for advances in their knowledge on such published literature as is generally available, and on necessarily limited contacts. Recently, however, as readers of the technical literature will be aware, there has been an awakening of interest in the proper organisation of microchemistry in this country, and the first fruits borne by this have taken the form of the microchemical meeting which was held in the Department of Applied Science, St. George's Square, Sheffield, on Saturday, October 9. This meeting was organised by Mr. R. Belcher, local representative of the Microchemical Club, in co-operation with the South Yorkshire section of the Royal Institute of Chemistry and the Sheffield Metallurgical Association.

Apparatus on View

In the morning, there was an exhibition and demonstration which included widely varied apparatus illustrating a good range of the various branches of microchemistry. An Oertling microchemical balance of the latest type (No. 63 PB.) with a 5-in. nichrome beam, was on view in position on a Griffin and Tatlock anti-vibration table. The complete combustion train for the estimation of sulphur and carbon in steels was set up, and there was, in addition, a specimen of the Griffin and Tatlock all-electric furnace for carbon and hydrogen estimations, and an electric muffle furnace and drier.

A Conway micro-burette (Gallerkamp) illustrated the principles used in volumetric apparatus delivering quantities of the order of 0.1 to 0.001 ml. In addition, there was a syringe burette based on the pattern recommended by Rosebury and van Heyningen,¹ operated by a micrometer plunger. This burette is used for titrating quantities of the order of half a ml. It was set up in conjunction with the special titration vessel of Benedetti-Pichler and Siggia,² which is constructed so as to give a large depth of colour for the observation of the indicator change, in spite of the small amount of liquid being handled. At the other end of the scale from these burettes, there were examples of Wigglesworth burettes and pipettes,³ which deal with quantities of liquid of the order of 0.3 to 0.03 cub. mm.

Electrometric apparatus was well represented. Both the Cambridge recording polarograph and the Cambridge non-recording voltamscope were on view. The Cambridge portable pH meter was fitted with a Stadie type electrode, which permits the use of very small quantities of sample. There was also a Cambridge titration apparatus for potentiometric work. Much interest was shown in the gas analysis apparatus, which was later described at length in one of the symposium papers.

In conjunction with a Spekker photoelectric absorptiometer, which was being demonstrated, there were charts showing the application of the instrument to the analysis of steels and other alloys. Finally, there were

several miscellaneous pieces of apparatus, such as a modified methoxyl apparatus, a semi-micro pressure calcimeter, and examples of various types of micro-desiccator. To round off the display, there was a selection of books and pamphlets dealing with a representative number of the various branches of microchemistry.

The Symposium

In the afternoon a symposium on Microchemical Analysis opened, with Mr. E. J. Vaughan, local chairman of the Royal Institute of Chemistry, in the chair.

The first paper, by Mr. C. Whalley, dealt with "Some Applications of Absorptiometric Methods to the Microchemical Analysis of Metal Alloys." Mr. Whalley explained that the work which he was about to describe arose through the numerous problems which came up nowadays in steel analysis, and which were incapable of solution through classical methods. By using a Spekker photoelectric absorptiometer, and employing filters and difference methods, subsequently modifying the methods to deal with micro quantities, it was found possible to carry out a micro analysis of steel for the alloy elements carbon, sulphur, silicon, phosphorus, manganese, nickel, chromium, molybdenum, vanadium, titanium and copper, more accurately than was possible on the macro scale, and using absorptiometric methods for all the elements but carbon and sulphur. Methods were also developed along the same lines for the elements iron, copper, nickel and manganese in aluminium alloys.

An accurate estimation of manganese, nickel, chromium and molybdenum can now be carried out on one 4 mgm. sample of steel, and it has been found that the total time required for the estimation of these four elements on six different test samples of steel is 4½ hours. Comparison of the results with similar analyses for these steels on the macro scale showed that the nickel varied by not more than 0.08%, and the other three elements by not more than 0.03% from the values found by the classical methods.

Micro-Volumetric Apparatus

Dr. G. H. Wyatt, in his "Review of Methods for Micro-titration," illustrated by means of slides a selection of the apparatus which is used for this purpose, although he pointed out that it was only possible to deal with a small fraction compared with the wide range which had been proposed in the literature from time to time.

Dealing first with a selection of useful graduated flasks, in some of which it is possible to carry out a number of operations without transference, he went on to illustrate a large number of pipettes, including the precision wash-out type, automatic pipettes of the Wigglesworth pattern, and devices for aiding delivery and for dealing with sub-micro quantities of liquid.

In his review of burettes, Dr. Wyatt dealt with both the horizontal and vertical patterns, and with means of controlling the delivery of the liquid. He also included several burettes of the piston type, and a typical weight burette. Finally, he illustrated types of vessels in which titrations could be carried, such as that recommended by Benedetti-Pichler and Siggia, and on display in the exhibition.

¹ *J. Ind. Eng. Chem., Anal. Ed.*, 1942, **14**, 363.

² *J. Ind. Eng. Chem., Anal. Ed.*, 1942, **14**, 828.

³ Wigglesworth, *Biochem. J.*, 1937, **31**, 1,719.

The Polarograph

Mr. J. T. Stock spoke on "Microanalysis with the Polarograph." Where there seems to be a general impression that exceedingly expensive special apparatus is necessary to carry out polarographic work, Mr. Stock pointed out that it is possible, if a suitable galvanometer is available, to carry out work of the non-recording type which will at least indicate clearly whether the polarograph is of any use in a particular problem. If it prove to be so, naturally advantages are to be gained with instruments of the recording type, in such directions as speed and the possession of a permanent record of an analysis. Although the bulk of the work with the polarograph has been on the inorganic side, there is an increasing amount of investigation of its use in the organic field.

A more recent development of polarographic technique has been in the line of amperometric titrations. This work, which was begun by Heyrovsky, has largely been developed by Kolthoff and his co-workers. In such work, since the applied potential is kept constant, the apparatus is somewhat simplified.

Estimations such as that of copper with α -benzoin oxime can be carried out in this way, and in a number of cases it is also possible to determine two substances in one solution, as is illustrated by the graph obtained when copper and zinc are titrated with quinaldinic acid.

Mercuric Oxycyanide

"The Use of Mercuric Oxycyanide as a Microchemical Reagent" was the subject of the paper by Mr. G. Ingram. He first reviewed the difficulties to be met with in the usual methods for estimation of sulphur. Mercuric oxycyanide was first introduced by Vieboch as a reagent in micro-halogen determination, the halogen releasing from the reagent an equivalent amount of hydroxyl ion, which can be determined by titration. By an extension it was found possible to determine halogen and sulphur in one sample, the total acids derived from these two elements being estimated, and the halide then estimated with the reagent, giving sulphur by difference. Further investigation has shown that it is possible to precipitate sulphate from sulphuric acid with barium chloride, and then to determine the excess barium chloride. This method of determining the sulphuric acid can of course be transferred to the micro-estimation of sulphur. The use of the reagent in estimation of ionic halogen has also been extended to cover the determination of methoxy and ethoxy groups by modifying the standard Pregl apparatus and utilising the alkyl iodides produced in the Zeisel determination. Thus it can be seen that the reagent has a use in a variety of determinations, with the added advantage that a number of errors which occur in the older methods are decreased.

Micro Gas Analysis.

The paper of Mr. W. B. Price, "A Rapid Method of Micro Gas Analysis," dealt with the apparatus which had been on view in the morning. This apparatus, which was developed by Krogh for investigating the gases dissolved in blood, was modified by Enss in 1932 to deal with bubbles enclosed in glass, the particular field to which the apparatus on view had been applied.

In the first stage the surface of the glass is ground away until the bubble is almost reached, and the glass is then immersed in a special box containing glycerine.

The bubble is pierced, and the gas is led out, using a dissecting needle, to the under side of a glass diaphragm covering the box. There its diameter is measured by means of a microscope, and the bubble is then removed by means of a special pipette to various reagent tubes of different pattern (according to the reagent). After treatment with each reagent the bubble is again taken back to the box and its diameter measured, any decrease being due to absorption of a gas.

It is possible, using these methods, to analyse within an hour a bubble of 0.2 mm. diameter containing all or any of the following gases: Sulphuretted hydrogen, carbon dioxide, oxygen, carbon monoxide, and hydrogen. Any residue is assumed to be nitrogen, since in this special use of the apparatus it is not considered necessary to look for organic gases. In addition, single tests have been devised for sulphuretted hydrogen, sulphur dioxide and water vapour. The last two of these gases cannot be determined in the ordinary way since they are soluble in glycerine.

Although the apparatus is not highly accurate, partly because of solution of the gases in the glycerine, and partly because the bubbles are measured on the assumption that they are completely spherical, which they are not, yet there is no doubt that valuable information can be obtained in this way; and there seems no reason why there should not be an extension of the methods to deal with other problems.

Teaching of Microchemistry

Dr. Cecil L. Wilson, who spoke on "The Teaching of Microchemistry," claimed that the definition of microchemistry should be interpreted as widely as possible, covering a field which was only in small part represented by the exhibition and by previous speakers. From experience, Dr. Wilson knew that many microchemical methods could be taught to students, so that the subject was not the closed field of a few experts. Many advantages were to be gained by giving students a six weeks' course, touching as many aspects of the subject as possible, whether they were going to use micro methods in their future career or not. However, he also felt that this was not sufficient. While a course which would be the equivalent of this six weeks' course should be available to all chemists or would-be chemists, there should also be refresher courses in various branches of the subject readily accessible, so that chemists could keep up with the advance in knowledge, or could obtain specialised training in some fresh branch. Dr. Wilson outlined very briefly a specimen general course, based on one which he himself had taught, and which covered qualitative and quantitative techniques in the inorganic and organic fields, and also what he termed "miscellaneous techniques," such as spectrography, chemical microscopy and electrography.

The Future of Microchemistry

After according a hearty vote of thanks to the speakers, the meeting went on to consider the present state and future organisation of microchemistry in this country. A number of speakers expressed the view, which was endorsed strongly by the meeting, that every effort should be made to advance microchemistry by proper organisation. In order to give effect to this, a Committee was elected by the meeting to investigate how best the science could be benefited by organisation, and to take appropriate steps to this end.

Converting Furnaces from Oil to Coal Firing

In view of limitations in the use of oil fuel and the need to conserve supplies for priority requirements, some metallurgical furnaces, previously oil-fired, have been, or are being, converted to pulverised-coal firing. The use of this latter method has been applied successfully for many years, but in this article reference is made to some recent conversions from oil to coal firing.

WAR conditions involve many changes in production technique because of the critical character of supplies of certain raw materials. For many purposes the use of a particular material under these conditions may not be a question of choice but rather of expediency. Such is the case respecting fuels and problems are being encountered due to the heavy demands for the raw materials for other purposes, or to difficulties encountered in supplying the quantities needed. In Britain the production of coal is an acute problem, not because of any scarcity of this important mineral, but because of the need for additional manpower to win it. Oil, on the other hand, must be imported, and since large quantities are used for other purposes the tendency is to convert furnaces from oil to coal firing.

It is apparent that the need to conserve oil fuel supplies is also creating problems in the United States, and to ensure that adequate supplies are available for more essential purposes many industrial heating units have been, and are being, converted from oil to coal firing, and some conversion methods applied to boiler and metallurgical furnaces are discussed by Hardgrove*; the latter are large users of fuel oil and present a considerable potential saving of fuel oil by conversion to pulverised-coal firing.

Pulverised-coal firing is applied with advantage in many plants operating in this country, but there are many circumstances to which the metallurgist must give careful thought when contemplating any departure from heating conditions as they may exist on his particular furnaces, the most important being the effect that another type of fuel may have on products. It is important, therefore, to consider what Hardgrove regards as the requirements of metallurgical furnaces fired by pulverised fuel, such as:—

1. Careful selection of coal as to its ash characteristics.
2. Very high fineness of pulverisation.
3. Uniform and controllable supply of coal and air to the furnace.
4. Furnace designs susceptible of being modified for satisfactory handling of the ash from the coal.
5. Flame with a high radiating characteristic and least tendency to oxidise the charge.
6. Conditions that will not increase the sulphur content of the metal being heated.

Metallurgical furnaces may be divided into two classes according to method of operation:—

"Dry" furnaces, in which the ash must be kept in a dry state, such as annealing furnaces, heating and forging furnaces, and soaking pits.

"Wet" furnaces, in which the ash can be in a molten state, such as malleable melting furnaces, copper reverberatory and refining furnaces, open-hearth, and some types of ingot or bloom-heating furnaces.

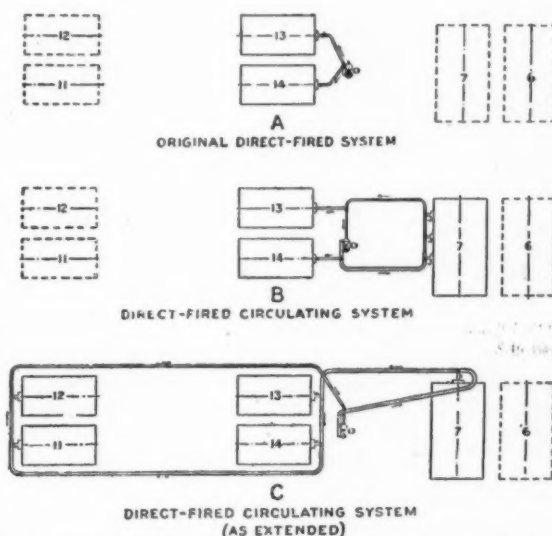


Fig. 1.—Direct-fired circulating system on malleable annealing furnaces.

The fact that many metallurgical furnaces have burned pulverised coal for years, and are still doing so, is proof that such difficulties as are inherent to the operation, and which are primarily concerned with the handling of the ash in the coal, can be overcome. Most of these earlier installations are fired by a storage system and the coal is pulverised with types of pulverisers that cannot obtain a high degree of fineness. Also the pulverised-coal feeders in the storage system increase the packing action which occurs in the storage bin, with the result that snowballing of the coal particles occur, which acts in the furnace in the same way as coarse coal. Many of the difficulties from slag particles on the product, and from sulphur contamination, are eliminated by very fine pulverisation. Furthermore, direct firing, which is being increasingly applied to metallurgical furnaces, not only eliminates all snowballing in the feeder and burner, but also removes this source of coarse coal in the furnace.

The selection of the most suitable coal is an important factor in the successful firing of metallurgical furnaces

* R. M. Hardgrove, *Mech. Eng.*, 65, 575-580.

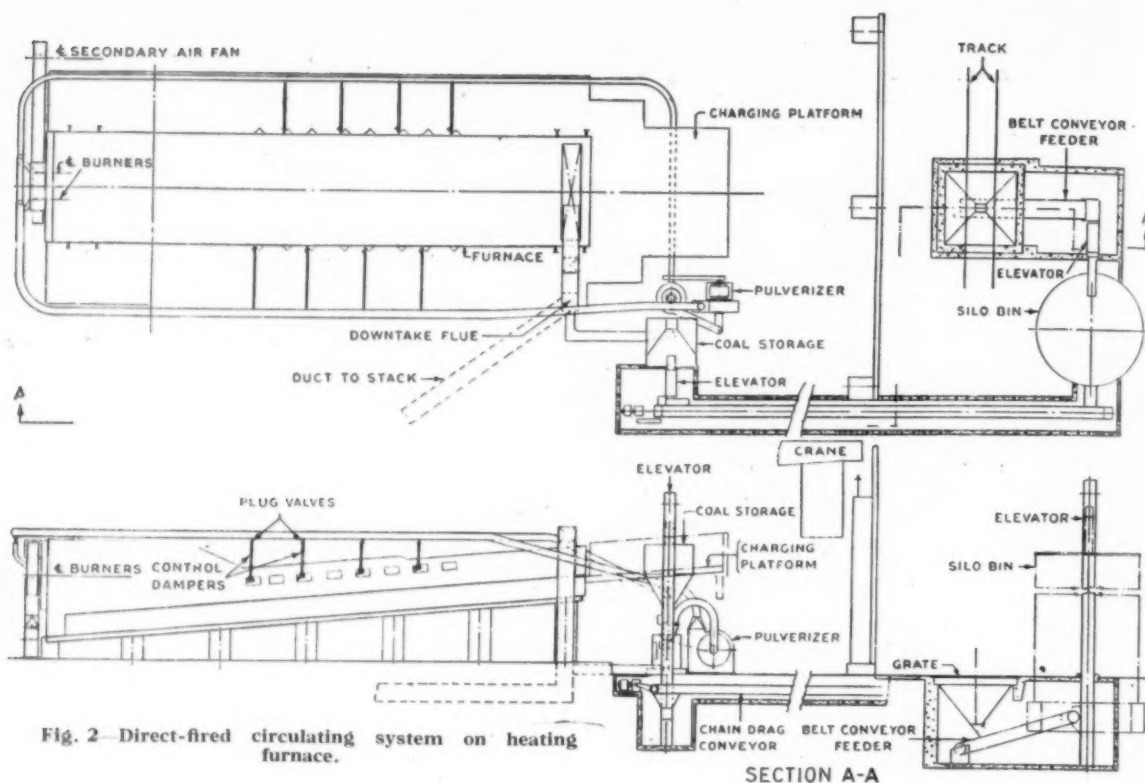


Fig. 2—Direct-fired circulating system on heating furnace.

with pulverised coal. High-fusing-ash coals are essential for dry heating furnaces operating at the higher temperatures where sticky ash is objectionable, but they are not necessary for furnaces operating at low temperatures, melting furnaces, and some furnaces which operate wet and tap out the slag. A medium- or low-sulphur content in the coal can also be obtained by careful selection; in fact, much lower than is often contained in certain types of fuel oil.

Copper reverberatory and refining furnaces have been successfully fired with pulverised coal for many years, and the same is true of forge furnaces, reheating and annealing furnaces. Continuous-billet heating furnaces fired with pulverised coal have been in operation for many years, and in some cases gas-heated regenerative furnaces for reheating have been converted to pulverised coal. The only change made in the complete furnace structure was to close up the gas-production section and to enlarge the apertures through the generative chambers.

It is probable that the malleable-iron industry has installed more pulverised-coal fire furnaces than other metallurgical industries, partly because the temperatures required are not high, and ash-handling problems are consequently simplified.

The development of an installation of pulverised-coal firing on annealing furnaces at a malleable-iron plant is shown in Fig. 1. The initial installation, shown at A, was a standard direct-fired system, serving two furnaces. The pulveriser had a greater capacity than was needed for these two furnaces, and as in their operating cycle they were off for considerable periods for charging, and were also operated at low rates during part of the cycle, the proposal was made to use the same pulveriser to

fire an additional furnace, No. 7, which was equipped with hand-fired grates. The direct-fired circulating system, as it was installed at that time, to take care of the three furnaces is shown at B.

In the direct-fired circulating system the piping forms a complete loop with the mixture of coal and air being tapped off at each furnace as needed, a portion of the coal and air being carried on past the last burner and returned to the base of the pulveriser, where it is mixed with the hot make-up air supplied to the pulveriser. In this particular installation the connections for each burner are made at the bottom of the loop-line, and a plug-type shut-off valve is located as close as possible to the main line. A butterfly control valve is placed below the plug valve, and the line arranged as nearly vertical as possible from the plug valve to the burner. This system keeps an ample circulation in this single-pipe system at all times, irrespective of how many burners may be in operation.

The installation shown in Fig. 1B was in operation 11 months, and proved to be so satisfactory that it was rearranged early this year and extended to serve five furnaces, as shown in Fig. 1C. There are seldom more than four of the five furnaces fired at one time, and there are periods when only one furnace is in operation. A second pulveriser is to be installed for the remaining five normalising furnaces in the plant with a similar direct-fired circulating system.

In a mill at one plant a billet-heating furnace has been converted to coal which previously was fired with a combination of oil and gas. The average consumption was about 2,400 gals. of oil per day, burned through two oil burners at the end of the furnace, each burner

having a maximum capacity of 60 gals. per hour. Sixteen gas burners were arranged along the sides, having a total maximum capacity of 5,870 cub. ft. of natural gas per hour. Because of space limitations it was impossible to locate a direct-fired pulveriser near the large burners, so that the pulveriser was installed at the other end of the furnace and the direct-fired circulating system arranged to supply the two main burners at the end and four small burners on each side.

The installation is shown in Fig. 2. This billet-heating furnace uses about 1,800 lb. of coal per hour at 40 grindability, and a fineness of 95% through 200-mesh. As any sticking of the ash to the billets in the furnace is objectionable, care was taken to obtain a suitable coal having a relatively high ash-fusing temperature and low ash and sulphur content. This installation has been in operation for a few months and the furnace conditions have been excellent, plain-carbon and low-alloy steel billets being heated uniformly and with much less scale than when heated with fuel oil.

Some sponge ash collects on most of the arch and falls to the floor, but this is not objectionable when the ash is dry. At one time several cars of coal of lower ash-softening temperature were received and the sponge ash on the roof became sticky. Upon dropping on the billets it gave some trouble in the rolling of the billets along the

hearth, but closer control of the coal supply has eliminated this difficulty. Also the underground flues were inadequate to eliminate a positive pressure in the upper end of the furnace, and the installation of hoods over the furnace openings has been necessary to stop fly ash spreading over the shop. With hoppers under each cleanout door to catch the ash removed during operation, or at the week-end shut-down, the ash removal is a satisfactorily clean operation.

Most furnaces designed for fuel-oil firing require some changes to take care of the ash from pulverised coal, but generally these changes are not extensive. When conditions permit, direct-fired pulverisers are the best means of firing pulverised coal to medium and large metallurgical furnaces. But there are many small plants which may be converted that have no space available for direct-fired pulverisers in the present building, and the direct-fired circulating system can be installed with the pulveriser outside the building. For firing a number of small furnaces, and particularly under crowded conditions, this direct-fired circulation system is especially suitable, but the fact that the cost of the single pulveriser for the latter system is less than the installation of direct-fired pulverisers should not be the deciding factor if it does not provide the best permanent operating installation.

Forthcoming Meetings

THE IRON AND STEEL INSTITUTE.

- Oct. 30. Joint Meeting of South Wales members and the Newport and District Metallurgical Society in the Newport Technical College, at 6 p.m. Mr. G. H. Latham, J.P., will preside and Principal C. A. Edwards, D.Sc., F.R.S., will present the paper by himself; Mr. D. L. Phillips and Mr. Y. H. Liu on "The Yield Point in Steel," original presented in May last at the Annual Meeting of this Institute.

INSTITUTE OF BRITISH FOUNDRYMEN BIRMINGHAM BRANCH.

- Oct. 30. "The Feeding of Non-Ferrous Castings," by W. Baker. At the James Watt Institute.

LINCOLN BRANCH.

- Oct. 30. "Running and Riser in Relation to Defective Castings," by E. W. Harding. Accompanied by a film on "Gating."
Nov. 13. "Radiography as an Aid to Foundry Technique," by W. Gladwell.

LANCASHIRE BRANCH.

- Nov. 13. "The Uses of the Syphon Brick Method of Cupola Tapping," by E. R. Dunning. At the Engineers' Club, at 3 p.m.

SCOTTISH BRANCH.

- Nov. 13. "An Atlas of Defects in Non-Ferrous Castings." Non-Ferrous Sub-Committee Report, presented by J. Arnott at the Royal Technical College, Glasgow, at 3 p.m.

WALES AND MONMOUTH BRANCH.

- Nov. 6. "Foundry Technique in Relation to Certain Pressure Castings," by F. Dunleavy.

W. RIDING OF YORKSHIRE BRANCH.

- Nov. 13. Works Visit to Messrs. W. C. Holmes and Co., Ltd., Huddersfield, at 3 p.m.

ELECTRODEPOSITORS' TECHNICAL SOCIETY.

- Oct. 25. "Zinc Plating from Sodium Zincate Solutions," by N. A. Tope, A.C.T.C., at the Northampton Polytechnic, E.C. 1, at 6 p.m.
Nov. 2. "Some Applications of Chromium Plating in Ordnance Manufacture," by D. D. Howat, B.Sc., Ph.D., at the James Watt Institute, Birmingham, at 5 p.m.

SHEFFIELD METALLURGICAL ASSOCIATION.

- Oct. 23. "The Education and Training of Metallurgists" A Discussion to be opened by Dr. Edwin Gregory.
Nov. 2. "Hardenability." Opening speaker: Mr. T. F. Russell, Dr. R. J. Sarjant, and Dr. W. Steven.
Nov. 16. "Metallurgical Aspects of the Welding of Hardenable Alloy Steels," by H. F. Tremlett, B.Sc.

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